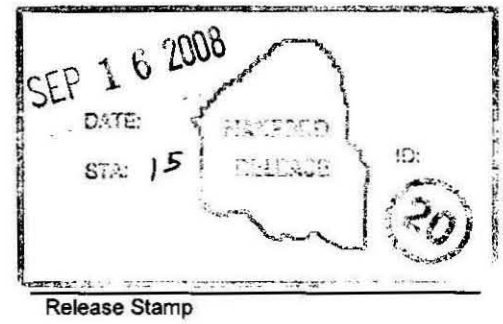
Hanford Medium/Low Curie Waste Pretreatment Alternatives Project - Fractional Crystallization Pilot Scale Testing Final Report

E. A. Nelson, D. J. Geniesse, and J. J. Zimmer
AREVA
Richland, WA 99352
U.S. Department of Energy Contract DE-AC27-99RL14047

EDT/ECN:	DRF	UC:	
Cost Center:	74A10	Charge Code:	
B&R Code:		Total Pages:	169

Key Words: fractional, crystallization, laboratory, tests, planning, pilot, simulated, tank, crystals, SRNL, waste, HLW, LAW, treatment, disposal, WTP, dissolved, saltcake, solids, pretreatment, design, construction, analyses, lessons learned, benchmark, baseline, hardware, centrifuge, pumps, heaters, instrumentation, configuration, controls, reboiler, steam supply,vacuum, solids, single-shell tank

Abstract: The Fractional Crystallization Pilot Plant was designed and constructed to demonstrate that fractional crystallization is a viable way to separate the high-level and low-activity radioactive waste streams from retrieved Hanford single-shell tank saltcake. The focus of this report is to review the design, construction, and testing details of the fractional crystallization pilot plant not previously disseminated.



R. D. Randal 09/16/08
Release Approval Date

Release Stamp

Approved For Public Release

RPP-39091
Revision 0

Hanford Medium/Low Curie Waste Pretreatment Alternatives Project – Fractional Crystallization Pilot Scale Testing Final Report

Date Published
September 2008

Prepared for the U.S. Department of Energy
Office of River Protection

Contract No. DE-AC27-99RL14047

Hanford Medium/Low Curie Waste Pretreatment Alternatives Project – Fractional Crystallization Pilot Scale Testing Final Report

Prepared for:
U.S. Department of Energy
Richland, Washington USA

Contract No. 25464
Project No. 01922.01.A501.02.11834

Prepared by:
AREVA Federal Services LLC
Richland Office
2425 Stevens Center Place – 2nd floor
Richland, WA 99354

Author:
EA Nelson, PE
DJ Geniesse
JJ Zimmer

Date Published:
August 2008

AREVA

Hanford Medium/Low Curie Waste Pretreatment Alternatives Project – Fractional Crystallization Pilot Scale Testing Final Report

Author: _____ Date: 8/28/08

EA Nelson, PE
AREVA Federal Services LLC

Author: _____ Date: 8/29/08

DJ Geniesse
AREVA Federal Services LLC

Approved: _____ Date: 8/28/08

JJ Zimmer
AREVA Federal Services LLC

TABLE OF CONTENTS

TABLE OF CONTENTS (contd)

TABLE OF CONTENTS (contd)

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES (contd.)

LIST OF ACRONYMS AND ABBREVIATIONS

ASV	Apparent Settling Velocity
CK	Centrifuge Cake
CPVC	Chlorinated Polyvinyl Chloride
CSD	Crystal Size Distribution
DAS	Data Acquisition System
DF	Decontamination Factor
DO	Draw-Off
DST	Double-Shell Tank
DTE	Draft Tube Entry
EDL	Engineering Development Laboratory
FA	Feed Tank A
FC	Fractional Crystallization
FCPP	Fractional Crystallization Pilot Plant
Free OH	Free Hydroxide
GTAW	Gas Tungsten Arc Welding
HLW	High Level Waste
HP	Horsepower
gpm	gallons per minute
ICA	Ion Chromatography for Anions
ICPMS	Inductively Coupled Plasma – Mass Spectroscopy
ICPOES	Inductively Coupled Plasma – Optical Emission Spectroscopy
IPS	Interim Pretreatment System
ITDP	Integrated Test and Demonstration Plan
LAN	Local Area Network
LAW	Low Activity Waste
mADC	milliamp direct current
NAS	Sodium (Na) Aluminosilicate
NPSHA	Net Positive Suction Head Available
P&ID	Piping and Instrumentation Diagram
PFD	Process Flow Diagram
PLC	Programmable Logic Controller
PD	Product Dissolver
PEDL	Process Engineering Development Lab
PLM	Polarized Light Microscopy
PTP	Pilot Test Plan
PSAL	Process Science Analytical Laboratory
PSE	Process Science and Engineering
QC	Quality Control
QEW	Qualified Electrical Worker
Q-UDS	Quick Undissolved Solids
rpm	revolutions per minute
RWT	Recycle Wash Tank
SR	Slurry Recirculation
SS	Stainless Steel

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

SRNL	Savannah River National Laboratory
SREL	Savannah River Ecology Laboratory
SST	Single-Shell Tank
SWT	Spent Wash Tank
TIC	Total Inorganic Carbon
TEASV	Trailing Edge Apparent Settling Volume
TRP	Test Run Plan
TS	Total Solids
UDS	Undissolved Solids
VFD	Variable Frequency Drive

HANFORD MEDIUM/LOW CURIE WASTE PRETREATMENT ALTERNATIVES PROJECT

FRACTIONAL CRYSTALLIZATION PILOT SCALE TESTING FINAL REPORT

1.0 EXECUTIVE SUMMARY

Fractional crystallization (FC) has been studied as a potential Hanford tank waste pretreatment process since January 2005. FC uses an evaporation and crystallization process to separate radioactive isotopes from the nitrate and nitrite salts that make up a large fraction of the waste in Hanford's tanks. As the liquid in the waste evaporates, salt crystals are left behind. These salt crystals form a matrix that generally excludes most radioactive isotopes, including cesium, technetium, and iodine. The overall testing program was developed to provide insights into how FC – a process similar to that used to purify table salt – could be used to separate the radioactive waste in the underground tanks at the U.S. Department of Energy's (DOE) Hanford Site into High-Level Waste (HLW) and Low-Activity Waste (LAW) streams. The LAW stream would be used to feed the Hanford Waste Treatment and Immobilization Plant LAW vitrification plant, reducing the volume of waste that must be treated as HLW.

Process modeling and flowsheet development was completed, followed by successful bench-scale testing using clean and real-waste simulants at the Georgia Institute of Technology (Georgia Tech) and actual tank waste at Hanford. The Engineering Development Laboratory (EDL) of the Savannah River National Laboratory (SRNL) assembled a Pilot Test Facility to achieve test conditions specified in the Process Flow Diagram (PFD) developed by AREVA Federal Services LLC (AREVA). Major components such as Crystallizer Vessel, Reboiler, Circulation Pump and Centrifuge were provided by Swenson Technology Inc. (Swenson). EDL designed and fabricated the balance of the system based on these documents and fundamental knowledge and experiences established in previous related experimentation.

Following construction activities, the EDL facility initiated a series of system checks and functional tests commensurate with the design of the facility and requirements for the safety inspection requirements for the facility and equipment. After completing all primary functional testing, the system was declared ready for chemical operations in April 2008.

In this large-scale (1/5th scale) pilot test, using non-radioactive simulants (including cesium) of the Hanford waste, SRNL successfully demonstrated FC's effectiveness. The EDL completed the seven-week pilot test of the FC technology as part of overall project testing and demonstration program described in the *Hanford Medium/Low Curie Waste Pretreatment Alternatives Project Integrated Test and Demonstration*

Plan. The pilot scale tests demonstrated that FC effectively separates non-radioactive sodium salts from cesium such that all cesium decontamination and sodium product yield goals were met. Specifically, the pilot:

- Achieved an Average Filter Cake Cesium Decontamination Factor of 130, compared to a goal of at least 50.
- Achieved a Sodium Product Yield (percentage of sodium isolated to send to LAW vitrification) of 52%, compared to a goal of 50%.

As with most Research and Development (R&D) activities, many challenges were faced during the pilot testing and the combined team of AREVA, Swenson and SRNL EDL overcame the obstacles to complete the required testing on time, within budget, and with results that met or exceeded the desired goals of the program.

2.0 INTRODUCTION

The Fractional Crystallization Pilot Plant (FCPP) was designed and constructed to demonstrate that FC is a viable way to separate the high-level and low-activity radioactive waste streams from retrieved Hanford single-shell tank (SST) saltcake. Details of the preliminary laboratory testing program that led up to the pilot testing phase are contained in documents referenced by the *Fractional Crystallization Pilot Scale Testing Preliminary Report*, RPT-3000559, Rev 000, and will not be repeated herein. The focus of this report is to review the design, construction, and testing details of the FCPP not previously disseminated.

2.1 Pilot Design

Design and fabrication responsibilities for the FCPP were divided between a team consisting of AREVA, Swenson, and the SRNL EDL group. The AREVA/Swenson team was responsible for delivering the crystallizer, reboiler, centrifuge and the main recirculation pump while the EDL group was responsible for installing these components along with required instrumentation and support systems in the SRNL 786A Building. The overall crystallization process is depicted in AREVA drawing C-01118-008, Revision 1, *Pilot Plant Crystallization System Process Flow Diagram*. For the purposes of this report, the FCPP, as shown in SRNL drawing EES-23164-M6-001, *Fractional Crystallization Pilot Scale Facility P&ID*, Revision "D", will be divided into four major subsystem groupings:

1. Crystallizer - The crystallizer subsystem consisted of the crystallizer vessel, recirculation pump, reboiler, condenser, steam jet vacuum pumps, and the interconnecting piping.

2. Centrifuge - The centrifuge subsystem consisted of the centrifuge, its programmable logic controller (PLC), and the 1½" slurry draw-off line that supplied the crystal slurry from and returned it to the 6" main crystallizer recirculation pipe.

3. Data Acquisition System (DAS) - The DAS consisted of the instruments and the LabVIEW™[1] software configured to monitor and control various process functions.

4. Balance-of-Plant (BOP) Systems - The BOP systems consisted of utilities, e.g. steam and electrical power, components, and piping systems necessary to interconnect all the FCPP process functions.

2.1.1 Crystallizer

The purpose of the crystallizer is to evaporate water from the incoming slurry (from the reboiler) until the dissolved salts reach a supersaturated condition. At this point, nucleation of solid salt crystals occurs and as long as proper conditions, i.e. time, temperature, and super-saturation prevail, the crystals will grow until they are of proper size for separation from the circulating slurry. The crystallizer operating temperature is fixed at a predetermined value established by a thermodynamic model of the system and is controlled by the head space pressure which is modulated by the vacuum system through an atmospheric bleed valve. The crystallizer boiling rate is fixed by the steam flow rate to the reboiler. This boiling rate is predetermined to create a vapor flow rate at the operating pressure that will effectively load the demisters. As evaporation and draw-off to the centrifuge lower the crystallizer liquid level, fresh feed is introduced to the recirculation loop upstream of the reboiler to maintain the liquid level. The feed rate should be nearly constant at steady state. A stable liquid level is required to maintain constant back-pressure on the draft tube to assure that boiling occurs in the crystallizer vessel and not in the recirculation loop or reboiler.

The slurry flows into the crystallizer through an internal draft tube with a conical outlet. The conical outlet promotes even radial distribution of the slurry to the boiling surface of the existing liquor volume within the vessel. This flow distribution ensures a uniform vapor release velocity, which is necessary to prevent splashing or slugging of liquid vertically into the entrainment separator and limits carryover of dissolved solids into the vapor stream. For experimental purposes, the draft tube may consist of three interlocking sections (one flared section and two, 1' straight extensions) which allow slurry volume changes for residence time adjustment. For the FCPP testing only the flared section was utilized, i.e. the operating volume provided the minimum crystal residence time.

The crystallizer is a stainless steel (SS) vessel 23' high with a major diameter of 5'-0" at its cylindrical mid-shell section and conical transition sections at both the top and bottom (see Northwest Copper Drawing D-7007, Appendix 8.3). A vortex breaker at the outlet nozzle prevents rotation of the fluid in the recirculation pipe leading to the recirculation pump. The crystallizer is supported by structural steel (pipe) columns to provide a minimum net positive suction head available (NPSHA) at the recirculation pump suction centerline.

[1] LabVIEW is a registered trademark for National Instruments, Austin, TX

Figure 1. FCPP Crystallizer, Reboiler and Recirculation Pump

2.1.1.1 Recirculation Pump

Slurry flow from the crystallizer through the reboiler and back to the crystallizer was provided by an axial flow recirculation pump. The axial flow pump was chosen to minimize damage to the crystals in the slurry. The operating flowrate (fixed at ~ 600 gpm) was established to minimize slurry temperature rise across the reboiler (less than or equal to 3°F for tube fouling considerations) while maintaining solids in suspension (velocity at ~6.5 fps) to prevent reboiler tube blockage. The recirculation pump was equipped with a variable frequency drive (VFD) that provided slurry flow rate, pump speed, and pump current readings to the DAS.

2.1.1.2 Reboiler

The SS reboiler was supplied with low pressure (~3 psia) saturated steam through a measuring orifice and a flow control valve. Since the steam becomes superheated as it is throttled to the reboiler shell pressure, condensate is sprayed through a desuperheater nozzle to remove the superheat and provide saturated steam to the reboiler. The low temperature rise and large heat transfer surface (~112 ft^2) prevent film boiling in the reboiler tubes.

2.1.1.3 Condenser and Steam Jet Vacuum Pumps

To meet the condensing capacity required for the design crystallizer vapor flow, a new SS condenser (Figure 2) was procured by SRNL for the FCPP from Hoffman Process, Inc. The unit was sized to condense 900 lbs/hr of vapor (at 131° F) while the expected crystallizer design flow was 684 lbs/hr. The condenser used SRNL Site process water for cooling (design flow 75,000 lb/hr @ 70° F inlet temperature) and was equipped with a pressure relief device attached to the vapor inlet piping.

Figure 2. Condenser During Installation.

The steam jet vacuum pump (Figure 3) used to remove non-condensable gases from the condenser and reboiler was a two-stage Schutte & Koerting Type AHTR steam jet ejector system. The air ejectors were capable of removing 25 lb/hr of air

and 3 lb/hr of water vapor at 2" Hg absolute when supplied with a total of 42 lb/hr of steam at 100 psig and 4 gpm of process cooling water at 85° F.

Figure 3. Two-Stage Air Ejector

2.1.2 Centrifuge

The Krauss-Maffei HZ – 40 Si centrifuge (Figure 4) was the solid/liquid separation device for removing product crystals from the draw-off loop (recirculating slurry side-stream). Due to a long lead time and cost for a new unit a factory reconditioned unit was purchased. The reconditioned unit was guaranteed to perform as well as a new unit however automatic basket speed control was not provided, i.e. speed was controlled by manually adjusting hydraulic pressure. The loop flow rate was determined by the minimum velocity (~ 6.5 ft/sec.) required to maintain the crystals in suspension in the 1½" draw-off pipeline while at the same time providing sufficient head to overcome the elevation difference between the location of the pump and the centrifuge slurry feed valve.

The draw-off flow from the crystallizer to the centrifuge was provided by a Flowserve 2K2x2R pump rated for 40 gpm at 36 ft. total developed head (TDH). A 2" pinch valve was installed at the slurry return to the main 6" crystallizer recirculation loop to control the draw-off loop flowrate and ensure positive pressure at the draw-off point. A VFD controller was installed on the pump to maintain line velocity and reduce pipeline pressure.

During the separation process, slurry was fed to a rotating basket where the crystals were retained on a screen (screen mesh size determines the minimum retained particle size) and the liquid (mother liquor) passed through the screen and was collected in a tank before recycling to the crystallizer or purging from the system. Therefore the draw-off rate was controlled by the centrifuge's capacity to process the incoming slurry.

Figure 4. Centrifuge Before Piping Installation

Factors affecting liquid flow through the cake include crystal size distribution (CSD), cake thickness, cake porosity, and centrifuge rotational speed. Excess fines (small crystals) in the slurry can clog cake interstitial spaces (pores) and impede liquid flow and therefore limit centrifuge processing capability. The centrifuge operational cycle (feed, wash, and cake peel) are controlled by the vendor-supplied PLC. During the peel step a small residual cake (heel) was left as a "bed" for the next slurry batch. Over a period of time the heel becomes packed or clogged by fines and must be removed to restore capacity. To prevent contamination of the product discharge chute, only condensate was used to dissolve the heel.

Product cake was discharged to the insulated Product Dissolver Tank (Figure 5) through a 4" vertical pipe and dissolved in condensate. Because crystal dissolution is an endothermic reaction, an electrically operated heater was employed to maintain process temperature at 131° F for the optimum dissolution rate.

Since the dissolver solution was used for washing the centrifuge cake, it had to be maintained near sodium nitrate saturation (~10M Na) to prevent centrifuge cake dissolution. At this concentration, sodium carbonate and sulfate are oversaturated. To remove these solids from the centrifuge wash solution, a cross-flow filter was installed on the product dissolver. Concentration of the dissolver solution was monitored by specific gravity of the solution and condensate was added to control the concentration at the near saturated condition.

Figure 5. Product Dissolver Below Centrifuge Platform

2.1.3 Data Acquisition System (DAS)

The Data Acquisition (and Control) System for the FCPP was composed of National Instruments® (NI) Signal Processing and Acquisition hardware and NI Labview® DAS software coupled with a Dell Workstation computer. Specifics of the DAS are included in Section 3.4.

The DAS was used for data acquisition and process alarms only. Process controllers were not installed in the DAS software. Therefore, process flowrates, temperatures, pressures, densities, vessel levels, and other control points had to be adjusted by operators by throttling manual valves.

2.1.4 Piping, Instrumentation, and Miscellaneous Components

AREVA/Swenson provided the PFD and sized the piping for the crystallizer recirculation loop. The detailed design for the FCPP piping was performed by SRNL and installed in accordance with guidelines provided by AREVA/Swenson and with the configuration depicted on the SRNL P&ID.

Process piping consisted of small diameter (¼ to ½") SS tubing and Swagelok fittings, ½" to 2" CPVC pipe and solvent welded fittings, and welded 6" and 8" schedule 10 SS pipe. Steam supply pipes were 1.5" schedule 40 threaded SS pipe with SS tubing branches. Threaded and solvent welded piping were selected to facilitate field fitting using existing supports wherever possible. The process piping was sized to provide reasonable pressure drops while maintaining line velocities to maintain solids in suspension. Numerous sample ports were provided as well as connections for flushing out lines with condensate to prevent and remove plugs when the systems had to be shutdown. No allowance was made for corrosion since the test would be short term.

Instruments used included flowmeters, thermocouples, tank level indicators, pressure and vacuum gauges and transmitters. Existing EDL instruments were used wherever possible. New instruments were specified and purchased when unavailable from the existing EDL stocks. Instrument data that was needed for process analysis was connected to the DAS and recorded electronically. Instruments that were expected to be useful only for troubleshooting or primary instrument backup provided local readout only.

Miscellaneous components such as small pumps, tanks, equipment supports, etc. were either selected from SRNL stock, or purchased new when suitable equipment was not available. Most of the pumps were simple centrifugal pumps with single mechanical seals. The draw-off pump was designed with double mechanical seals and a pressurized seal water supply. A special centrifugal pump with a flow inducer was provided to pump condensate from the reboiler while preventing cavitation at low pressure.

To ensure an adequate steam supply to meet the expected demand from the reboiler and the air ejectors, a new Electro-Steam 250 KW package electric steam boiler was installed in parallel with the existing 150 KW unit. Both boilers were connected to a common 1½" header and cycled on-and-off in response to header pressure. Each unit had their own integral, independent control system and an installed relief valve for over-pressure protection. Process condensate was supplied to the boilers for feed water make-up.

Polypropylene tanks were used for in-process tanks. The heated and insulated centrifuge product and wash solution tanks, along with typical heater controls, automatic valves, instruments, and pump, etc. are shown Figure 6.

Figure 6. Centrifuge Tanks and Miscellaneous Controls

Large polypropylene tanks were used to store the feed and recycled simulant solution. The feed tanks are depicted in the P&ID and shown in the "tank farm" picture (Figure 7). The tanks were wrapped in plastic film as a precaution against a spray rupture event. The tanks were installed in a 1-foot high flexible secondary containment around the tanks to guard against a spill release to the environment.

Figure 7. Tank Farm

Excess condensate from the process was stored in covered, plastic tanks outside the EDL. During Construction Acceptance Testing all tanks were "calibrated" by pumping in known volumes of water then measuring level rise. Where applicable, the calibrated levels were compared to indicated levels from ultrasonic level detectors. All tanks were equipped with high level alarms that alarmed at the Shift Engineer's desk.

2.2 Pilot Plant Construction

Pilot plant construction was performed by EDL staff and SRNL Site construction craftsmen. Prior to installing the FCPP systems and equipment, some modifications were required for the 786A Building including the removal of materials from previous testing programs and strengthening support structures. Construction activities, including Construction Acceptance Testing, commenced in July, 2007 and completed in April, 2008.

2.2.1 Materials

The major equipment (crystallizer, reboiler, axial flow pump, and primary condenser) were constructed using 304-L SS. For the other equipment and piping, CPVC and polypropylene were selected for their ease of installation, the flexibility to rework if necessary, and adequate chemical and thermal resistance for use with the process chemicals. Pumps were generally SS both for better durability and because of their availability from the existing EDL equipment stocks. Graphite filled flexitallic gaskets were used between SS flanges; 1/8" thick viton rubber gaskets were used any time a CPVC flange was bolted to either a SS or another CPVC flange. Small diameter polyethylene tubing was used to pipe condensate streams because it was easy to route and adequate for the expected process temperatures and pressures. 316 SS tubing was generally used for process lines less than ½" in diameter.

2.2.2 Installation and Testing

The 786-A building was high enough to enclose the crystallizer and reboiler. However, minor structural modifications were required to accommodate the new components weighing 3 tons and 1 ton (dry weight), respectively.

Major equipment was placed in the building according to a preliminary assembly drawing prepared by the EDL staff. Large SS piping was detailed on drawings and shop fabricated in sections, with the final fit and welding in the field. Minor equipment, instruments, and piping less than 6" was field installed to meet the requirements shown on the P&ID. Standard Unistrut was used to support instrumentation and piping. Existing building steel was used, where available, to minimize the need for customized supports.

Major equipment and tanks were surrounded by 12"-18" high flexible secondary containers sized to hold the entire contents of the largest single source of chemical spills or leaks.

Electrical connections were utilized from existing EDL 120V single phase and 208V three phase receptacles. Three 480 V three phase disconnect/receptacles were installed to power the 15 HP axial flow recirculation pump, the 10 HP draw-off loop pump, and the 7.5 HP centrifuge.

Piping was installed and tested in accordance with the ASME B31.3 process piping code. For testing purposes, the process chemical piping was judged to be "category normal" while steam, condensate, and air were categorized as B31.3 "category D." Welding of SS pipe was performed by machine shop staff formally trained and qualified on the GTAW process. The EDL technicians were formally trained and qualified for solvent welding of CPVC piping. An independent inspector visually inspected over 5% of the GTAW welds and witnessed over 5% of the solvent welds on the process chemical piping. The process pipes were hydrostatically tested according to specific written work instructions as specified in the code. Wherever possible, the non-code piping was included in the hydro test of the code piping. However, sections that could not be pressurized (such as piping between open top tanks and the first valve) were leak checked during initial system shakedown with water.

Many of the instruments were bench calibrated before installation. Some that were provided simply for troubleshooting were not calibrated while a few of the instruments had to be calibrated *in situ*. The master instrument list identified in Appendix 8.5 identifies which instruments were calibrated.

2.2.3 Manufacturer's Instructions

Manufacturer's Instructions covering installation, operation, and maintenance of certain FCPP equipment were provided to SRNL for inclusion in Work Instructions. These instructions included:

- Crystallizer Operating Instructions (Swenson)
- Gould's Model AF (6x6-6") MXR Pump (Goulds Pumps)
- KMPT HZ 40 Si Peeler Centrifuge (KMPT)
- Centrifuge Functional Design Specification (KMPT)
- Pump Smart PS 200 drive (ITT)

2.2.4 Readiness Certification

In preparation for the chemical operations of the FC system, a management review was initiated to ensure proper documentation, training, and concurrence had been completed to ensure the facility was in the required readiness to operate safely and effectively. A Readiness Certification document was generated to establish the state of readiness to perform the R&D activities for the pilot-scale facility and is included as Appendix 8.9. The purpose of the readiness report was to establish a documentation record for the readiness of the facility for chemical operations based on the following criteria: Work Construction, Safety Compliance Documentation and Controls, Compliance with Conduct of Research and Development, Work Documentation Completion and Readiness, Training Status for Personnel,

Compliance with Expectations from Customer, Satisfying Internal Management Requirements, Satisfying DOE Oversight. A thorough review was completed and documented in the report and approved by the EDL management, the Manager of Research Programs and the Manager of Safety Programs within the SRNL.

3.0 TEST PLANNING

Testing of the FCPP was the last event in the *Hanford Medium/Low Curie Waste Pretreatment Alternatives Project Integrated Test and Demonstration Plan (ITDP)* RPP-PLAN-34134, Rev. 0. While the *Fractional Crystallization Test Plan,* RPP-PLAN-34135, Rev. 0 (Pilot Test Plan) contained specific test requirements at the pilot plant operations level, the ITDP incorporated requirements at a higher, program level. Specifically, these requirements involved validating or resolving certain enabling assumptions and uncertainties. In addition, contractual process performance requirements would have to be met and these would be validated by analyzing the process and product streams detailed in a laboratory Analytical Test Plan.

Significant enabling assumptions in the ITDP included:

- Modeling can be used to predict FC process performance with Hanford waste.
- Simulant can be used to investigate FC performance with actual Hanford waste.
- The FC process is scalable, i.e. the full-scale process performance can be predicted from laboratory, engineering and pilot scale operations.

ITDP uncertainties included:

- FC ability to handle feed variability.
- FC efficiency of separating salt crystals from the mother liquor containing cesium (Cs), technetium (Tc), and iodine (I).
- Crystallizer vessel retention time adequate for crystal growth.
- Complexity of the required FC control system necessary to provide ease of operation.
- FC system's ability to recover from process upsets.

Process performance requirements included removing at least 50% of the incoming feed stream sodium content and attaining a product DF of at least 50.

3.1 Pilot Test Plan

The Pilot Test Plan (PTP) incorporated the above assumptions and uncertainties to be addressed with the exception of modeling and use of simulants. Both the ability to effectively model the process and the comparison of simulant to actual waste

performance had been demonstrated in earlier project reports so they were considered "verified" in regard to further exploration during pilot plant operation. The PTP was structured to sequentially address the remaining assumptions and uncertainties in a series of five tests:

- Test 1 – Benchmark Testing
- Test 2 – Baseline Testing
- Test 3 – Process Parameter Variation Testing
- Test 4 – Feed Variability Testing
- Test 5 – Casualty Testing

Each test would be governed by a "Test Run Plan" (TRP) document that provided the operators with instructions on how to conduct the test, parameters to observe and data to be collected. As noted in the *Fractional Crystallization Pilot Scale Testing Preliminary Report*, RPT-3000559, the testing period was curtailed so that results could be utilized to select the technology for conceptual design of the Interim Pretreatment System (IPS). As a result of the accelerated testing schedule and a lack of additional funding, only Test 1 and Test 2 were completed. However in performing these test runs, some elements of the planned subsequent tests were also successfully demonstrated. Copies of the final Test 1 and Test 2 TRPs as implemented are contained in Appendices 8.10 and 8.11.

3.2 SRNL Analytical Test Plan

The final analytical plan was issued as document SRNL-PSE-2008-0046, Rev. 0 on April 16, 2008. This document outlined the analytical techniques to be performed and the expected sampling and analysis frequencies.

3.2.1 Sampling Frequency

The samples that were planned to be taken and the times at which these would be taken are shown in Table 1. The sample analysis techniques abbreviated in Table 1 are described in Table 2. The sampling described in Table 1 was roughly followed throughout both the Benchmark and Baseline phases of operation.

Initially, the destination tanks for the HLW and LAW fractions were misidentified (including in the issued Analytical Plan). A corrected flow diagram of sample locations was produced on May 8, 2008 and is shown in Figure 8. The "Purge Liquor" identified in Table 1 was meant to be the HLW product sent to HLW Receipt Tank E, but was incorrectly assumed to be the Liquor sample taken from the line from the centrifuge to the SWT during the dewatering step. However, the intended samples were actually taken periodically during the tests; these samples were identified as sample PR or PR(E).

Table 1. Sampling Locations and Frequencies Planned

Stream	Process Stage Or Time	Notes	Total Solids	Total Solids, Undissolved Solids (wt%)	Quick UDS (wt%)	Density	CSD	PLM	Apparent Settling Volume/ Density	Aluminum by Turbidity	Sample Prep	Free Hydroxide, Total Base	ICPMS Cs	ICP-OES	IC Anions	TIC/ TOC
Feed Tank	before start of run	reconstituted feed: archive only unless specific reason to analyze	X	X		X						X	X	X	X	X
Slurry Recirc	Prior to nucleation					Every 8hr										
	Nucleation to 10 wt% UDS				every 8 hr				every 8 hr							
	10 to 20 wt% UDS				every 2 hr				every 2 hr							
	20 wt% UDS to Steady State (~30 wt% UDS)			every 2 hr	every 1 hr				every 1 hr							
	Steady State						every 8 hr	every 8hr	Every 4 hr							
Slurry Draw-Off	Steady State		1 per Test				as needed	as needed								
Purge Liquor	1 at start and 1 at end of steady state portion of each Test		2 per Test								immediate dilution	X	X	X	X	X
Spent Wash Tank	1 per Test		X								immediate dilution		X	X	X	X
Cake 3	1 per Test (1, 2)						as needed	X		as needed during centrifuge tests	dilution		X	X		
LAW Product Tank	1 per day or per "batch" (c)	analyze 1 sample only per Test	X	X		X							X	X	X	X
Condensate	once per day	archive; analyze only if warranted											X	X		

Table 2. Abbreviations of Sampling Methods

Method	Abbreviation
Inductively Coupled Plasma – Optical Emission Spectroscopy for elemental analysis	ICPOES
Inductively Coupled Plasma – Mass Spectrometry for Cs	ICPMS
Ion Chromatography for Anions	ICA
Total Solids	TS
Undissolved Solids	UDS
Polarized Light Microscopy	PLM
Crystal Size Distribution	CSD
Free Hydroxide	Free OH
Trailing Edge- Apparent Settling Volume	TEASV
Apparent Settling Volume	ASV
Aluminum by Turbidity	-

To facilitate recombining LAW and HLW to reconstitute the feed, the LAW product could be sent to any of the feed/product tanks (A, B, C, D). These product samples were identified as FA, FB, FC, or FD as were feed samples from the same tank.

Samples were taken during the Baseline test from the Recycle Wash Tank and the Product Dissolver for various analyses. Samples from the centrifuge discharge lines to the SWT and RWT were also taken during the Baseline test. None of these samples were originally in the Analytical Plan.

3.2.2 Sample Types and Analyses Performed

The types of samples taken and the analyses typically performed on each type of sample are shown in Table 3. There are differences between these actual sample analyses performed and those that had been originally planned. Both the originally planned analyses and the actual analyses performed are discussed herein.

Occasionally, additional analyses other than those shown in Table 3 were performed. Numerous samples were analyzed for only density or density, total solids, and undissolved solids. The analysis method abbreviations are shown in Table 2. No samples were taken from the Draw-Off tap on the slurry feed to the centrifuge.

All samples were submitted for one of each analysis. Replicate samples were not submitted except in cases where the validity of results was in question. However additional samples were archived in case repeat or additional analyses were needed. Duplicate analyses at the laboratories were performed and the average concentrations determined were reported.

Table 3. Sample Types and Analysis

Sample Description	Sample Location	Abbreviation	Sample Preparation Method (1)	Sample Analyses
Feed	feed tank A or B bottom drain or dip sample	FA, FB	none	density
			none	density, ICPOES, ICPMS, ICA (+ initial & final samples: TS, Free OH, Carbonate)
Crystallizer slurry (Slurry Recirc)	slurry recirculation line	SR	none	density, TS
			hot filtration – solids	PLM
			acetone wash of solids and drying	CSD
			hot filtration – liquid	density, TS (to give UDS)
			none	TEASV or ASV
Crystallizer salt slurry @ draw-off line	draw-off line drain	DO	no samples taken	
Crystallizer condenser condensate (Condensate)	primary condensate line	CO	none	density, ICPOES, ICA, pH
Centrifuge liquor composite from peels 1-4 (taken 5/30 @ ~13:00)	line from centrifuge to SWT	LQ-0530-1300-B	none	density
			dilution; equal vol. each peel combined	ICPOES, ICPMS
Centrifuge liquor composite from peels 5-8 (taken 5/30 @ ~13:00)		LQ-0530-1300-E	none	density
			dilution; equal vol. each peel combined	ICPOES, ICPMS
Spent Wash Tank liquid	SWT tank dip sample	SW	none	density
			none	density, ICPOES, ICPMS (+ final sample only: TS, ICA, Free OH, Carbonate)
Spent Wash Tank washes 1-4 composite sample from Centrifuge (taken 5/30 @ ~16:00)	line from centrifuge to SWT	SW-0530-1300-(C1-C4)	none	density
			dilution; equal vol. each wash combined	ICPOES, ICPMS
Spent Wash Tank washes 5-8 composite sample from Centrifuge (taken 5/30 @ ~16:00)		SW-0530-1300-(C5-C8)	none	density
			dilution; equal vol. each wash combined	ICPOES, ICPMS
Recycle Wash Tank liquid (slurry)	RWT tank dip sample	RW	none	density
			dilution	ICPOES, ICPMS (+ final sample only: TS, ICA, Free OH, Carbonate)
Recycle Wash Tank washes 1, 2, 4 composite sample from Centrifuge (taken 5/30 @ ~16:00)	line from centrifuge to RWT	RW-0530-1300-C124	none	density

Sample Description	Sample Location	Abbreviation	Sample Preparation Method (1)	Sample Analyses
(wash 3 was missed)			dilution; equal vol. each wash combined	ICPOES, ICPMS
Recycle Wash Tank washes 5-8 composite sample from Centrifuge (taken 5/30 @ ~16:00)	line from centrifuge to RWT	RW-0530-1300-(C5-C8)	none	density
			dilution; equal vol. each wash combined	ICPOES, ICPMS
Product Dissolver Tank slurry	PD tank dip sample	PD	none	density
			dilution	ICPOES, ICPMS (+ final sample only: TS, ICA, Free OH, Carbonate)
		PD(F)	hot filtered – filtrate; diluted	ICPOES, ICPMS, ICA
		PD(S)	hot filtered – solids; diluted	ICPOES, ICPMS, ICA
Centrifuge Cake (product to PD)	chute to PD	CK	dilution	ICPOES, ICPMS
			none	PLM
HLW Product (from SWT)	HLW Tank E (2) bottom drain or dip sample	PR or PR(E)	none	density
			none	density, ICPOES, ICPMS, ICA (+ final sample: TS, Free OH, Carbonate)
LAW Product (from PD)	Tank C bottom drain or dip sample	FC	none	density
			none	density, ICPOES, ICPMS, ICA (+ final sample: TS, Free OH, Carbonate)

(1) Sample preparation method does not include additional dilution or acidification done by analytical laboratories prior to analyses.
(2) HLW Tank E was incorrectly called the Product Receipt Tank in "Analytical Plan for the Fractional Crystallization Pilot Plant", so the designation was PR or PR(E) to emphasize which tank was to be sampled

3.2.2.1 Density

Density measurements were performed on both clear (no undissolved solids) aqueous and slurry samples. Samples from every tank were analyzed. Initially, the density of clear samples was measured using an Anton Paar DMA 4500 density analyzer per ITS-WI-0029 or by weighing a known volume of sample in a precision volumetric flask. During testing operations, it was determined that there was insufficient time to complete all clear liquid densities with the Anton Paar analyzer, so all density measurements were switched to the volumetric flask method.

The Anton Paar analyzer was calibration-checked with deionized water at 25°C or 55°C. The mark on the volumetric flask was based on a fixed temperature of 20°C. All density measurements by volumetric flask were made with the sample starting at the process temperature; no attempts were made to control temperature during density measurement. A small error (<0.05%) in density for samples above 20°C was unavoidable but not significant. All density measurements were conducted by PSE staff at EDL.

3.2.2.2 Total Solids, Undissolved Solids (TS, UDS)

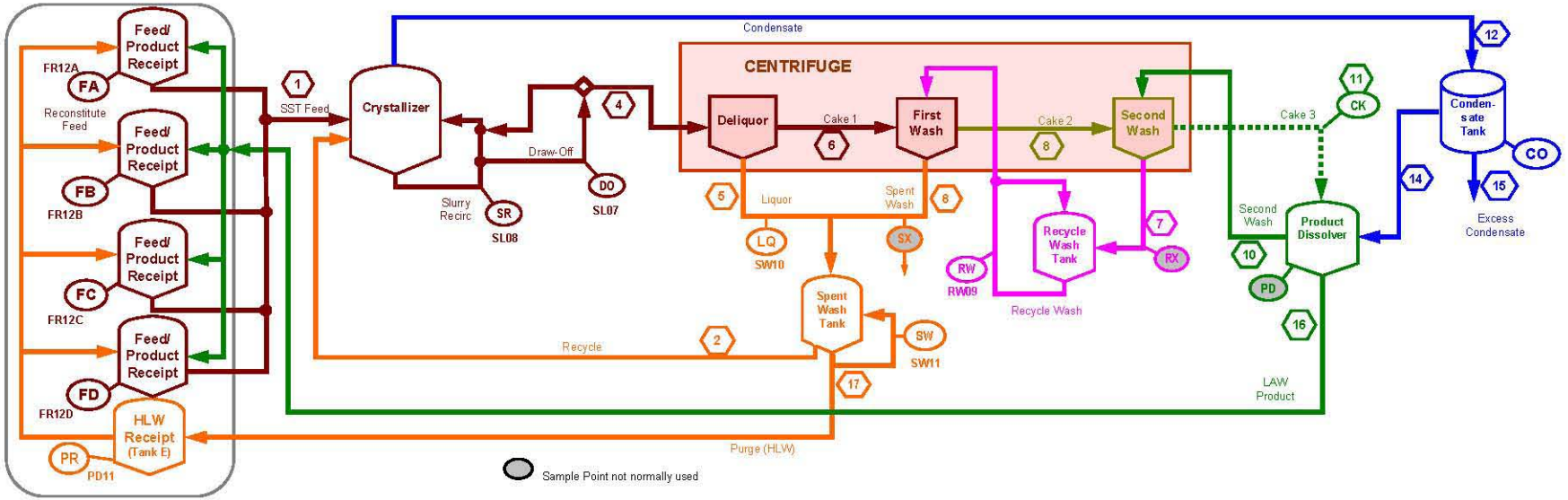
Total Solids is the weight percent of the total amount of solids, both dissolved and undissolved, that remains after the sample has been dried at approximately 150°C. Volatile components of the sample are not accounted for by this method. This analysis was performed using an AND MX-50 Halogen Lamp Moisture Analyzer per ITS-WI-0029. The analyzer was periodically checked by measuring the solids content of a standard NaCl solution. All solids analyses were conducted by PSE staff at EDL.

Undissolved Solids is the weight percent of the solids that are not dissolved at the conditions at which the sample is filtered. The UDS was measured on slurry samples using the following steps. First, the Total Solids content of the original sample was measured. Another sample, taken at the same time, was kept at the process temperature and hot filtered (in the same way the crystals are prepared for the CSD and PLM measurements). The filtrate was then analyzed for its total solids content, which was called the Supernate Solids, or SS. This filtrate did not need to be kept at the process temperature because the total solids content was independent of temperature. (There will be precipitation of undissolved solids, but this has no effect on the total solids measurement.)

The UDS was then calculated from TS and SS:

$$\mathrm{UDS} = 100\,\frac{\mathrm{TS} - \mathrm{SS}}{100 - \mathrm{SS}}$$

Figure 8. Flow Diagram of Sampling



3.2.2.3 Quick Weight Percent (Wt%) Undissolved Solids (Q-UDS)

A quick estimate of the UDS was performed on some Slurry Recirculation samples by filtration of the hot slurry. The sample was taken into an insulated container and then quickly filtered using a 350-mL medium glass frit Buchner filter (Chemglass CG-1402-23). The weights of the filtrate and cake collected were then determined from the final and initial weights of the filter and the filter flask. This method is known to be biased high due to the residual moisture in the filter cake. This process is described in ITS-WI-0026. These measurements were performed by PSE personnel at EDL.

$$\text{Quick Wt\% UDS} = \frac{(\text{Total Mass of Slurry} - \text{Mass of Filtrate})}{\text{Total Mass of Slurry}} = \frac{\text{Mass of Filter Cake}}{\text{Total Mass of Slurry}}$$

3.2.2.4 Trailing Edge Apparent Settling Volume / Density (TEASV or ASV/Density)

The Trailing Edge Apparent Settling Volume (TEASV) and Density were measured by placing a known amount of a Slurry Recirculation sample into a graduated cylinder with volumetric markings. This graduated cylinder was externally heated to maintain the process temperature of nominally 55°C. These measurements were performed per ITS-WI-0023.

A camera was used to photograph the graduated cylinder at 10 minute intervals for up to four hours. The volume of the settled solids and the total sample volume as a function of time were then determined by manually reviewing the recorded photos. The accuracy of reading the volume in the photos was about ±20 mL; the total volume was typically 180-240 mL and the final settled volume ranged from 130-210 mL. It was often difficult to determine the location of the actual interfaces between the air and sample and between the settled solids and the clearer liquid. For many samples, there was a foam layer on top of the liquid that was up to 60 mL volume. The settled solids interface was difficult to determine because this interface was often very indistinct.

The data recorded were entered into spreadsheets to produce plots of settled volume / total volume versus time. After about two weeks of operation, it was obvious that the TEASV curves were linear over the range measured, so only the last data point was plotted for many samples. These measurements were performed by PSE personnel at EDL.

3.2.2.5 Crystal Size Distribution

CSD was measured by separating the dried crystals into size ranges using sieves and a sieve shaker. The sieve shaker used was a Fritsch Analysette Pro 3. The sieves used were Newark stainless steel 3" sieves. The sieve sizes are shown in Table 4.

Table 4. CSD Sieve Sizes.

Sieve Number	Nominal Sieve Opening (μm)
20	850
30	600
40	425
50	300
70	212
100	150
140	106
200	75
270	53
400	38

The samples were prepared by hot filtering the Slurry Recirculation sample, followed by washing of the filter cake with acetone. The amount of acetone used was about four times the volume of the filter cake. The acetone washed cake was then vacuum dried for 12-24 hours at about 28 in. Hg vacuum. The vacuum drying was followed by oven drying at 110°C for several hours. The dried crystals were then gently separated and sieved through an 850 μm screen to remove the larger agglomerated particles. The resulting sample was then split evenly into eight fractions using a riffler device. Several sample fractions were then combined to give about 15g total sample. This sample was then sieved in the shaker. The sieving process is described in detail in ITS-WI-0025. The amounts of sample collected on each sieve were then used to produce a crystal size distribution plot. This method was performed by PSE staff at EDL.

3.2.2.6 Polarized Light Microscopy (PLM)

PLM was conducted per methods developed at Hanford (HNF-11585, Rev. 0). The microscope used was an Olympus SZX Research Stereomicroscope System outfitted with polarizers, a Red I compensator, and a Pax-It camera and analysis software. Numerous photos of various process samples were taken throughout the tests. PSE and Materials Science &Technology staff performed all PLM work.

3.2.2.7 Aluminum by Turbidity

The Aluminum by Turbidity method for determining the amount of aluminum in centrifuge cake samples was used for 11 cake samples and also for four feed tank samples. This method was not used after 5/17/08 because it appeared to be too insensitive to changes in Al concentration and the amount of Al in the cake wash solution from the Product Dissolver was higher than it would be after the process reached steady state with respect to Al.

3.2.3 Analyses Performed by Savannah River Ecology Laboratory (SREL)

3.2.3.1 Standard and Sample Preparation

Calibration and Quality Control (QC) solutions were prepared daily from certified stock solutions of single elements and/or custom multi-element blends (Inorganic Ventures, Fisher, Spex CertiPrep, Ultra Scientific and SCP Science). An internal control verification (ICV) created from a different certified stock solution and/or lot as well as blanks were included in daily routine analysis. The calibration and QC solutions were diluted with >18 $m\Omega/cm^3$ water (Barnstead NANOpure Diamond Water Purification System) and stabilized in 1% v/v ultra high purity HNO_3 (Fisher Scientific Optima Grade) when necessary. Samples diluted for Ion Chromatography analysis did not include HNO_3 as it interfered with the analysis. QC protocols were followed with different and same dilution replicates, blind standard checks, and spikes at 10 to 20% the original number of samples.

3.2.3.2 Free Hydroxide and Other Base Excluding Carbonate

3.2.3.2.1 Instrumentation

A Radiometer Analytical TitraLab 870 titration workstation was used with a Radiometer combined pH electrode.

3.2.3.2.2 Method

The Free hydroxide and other bases excluding carbonate determination were adopted from Manual L16.1 Procedure ADS-1206 revision 4.

100 μL of sample was transferred into a centrifuge tube containing 2 mL of H_2O. Approximately 2 mL of 0.5 M $BaCl_2 \cdot 2H_2O$ was added and sample was allowed to mix on a Vortex mixer for approximately 30 seconds. The sample was then centrifuged for approximately 2 minutes at ½ maximum speed to separate the precipitate. The supernate was then transferred quantitatively into 25 mL deionized water in a titration sample cup. These prepared samples were then titrated to pH 4.0 against a 0.100N HCl at a 0.1 mL/min or 0.01 mL/min in a dynamic IP mode, which controls the volume of titrant so that the increment size is inversely proportional to the slope of the titration curve.

Two inflection points were recorded: the first between pH 11 and 8 was equated to the free hydroxide normality; the second inflection point closest to 7.0 was considered to be the normality of the free hydroxide plus the other base excluding carbonate.

3.2.3.3 Carbonate by Total Inorganic Carbon (TIC) Measurement

3.2.3.3.1 Instrumentation

A Vacuum filtration apparatus and a vacuum extraction line for carbonate digestions were used for carbonate analysis.

3.2.3.3.2 Method

Fifty grams of saturated $BaCl_2*2H_2O$ solution is added to 20 grams of sample solution under a CO_2-free atmosphere to precipitate a Ba-salt that contains carbonate. After 12 hours, the solution is tested by adding a few extra drops of Ba-solution. If no new precipitate is observed the solid may be separated by vacuum filtration using a pre-weighed filter (0.2 micron) and dried to constant weight. The mass of solid is determined by weight difference and the solid is then homogenized with a mortar and pestle. An aliquot of known weight (~10 ± 0.005 mg) is loaded into an Inconel boat and placed in the sidearm of a common acid bath extraction vessel which is attached to a vacuum extraction line. The sample is digested in orthophosphoric acid, the resulting CO_2 is purified cryogenically, and measured manometrically to compute the number of moles of CO_2 generated by acid digestion. From this measurement, the percentage of C in the aliquot can be determined accurately and precisely (+/- 1%) and related to the mass of C in the original liquid sample.

3.2.3.4 Inductively Coupled Plasma- Mass Spectrometry (ICPMS)

ICPMS was used to measure only non-radioactive Cs (^{133}Cs).

3.2.3.4.1 Instrumentation

Cesium analyses were performed using a Perkin-Elmer Sciex Elan DRC Plus inductively coupled plasma mass spectrometer (ICP-MS, Norwalk, CT) in standard operating mode. The sample introduction system was chosen based on the difficulty of the sample matrix (high dissolved solids and low detection limit requirements). Samples were introduced using a Perkin-Elmer AS91 autosampler, Gilson Minipuls 3 peristaltic pump, and a Twister cyclonic spray chamber coupled with a Sea Spray nebulizer (Glass Expansion, West Melbourne, Australia).

3.2.3.4.2 Method

Germanium, Yttrium, and Indium were used as internal standards to check for instrument drift and matrix effects on all samples, standards, and blanks. All solutions were manually spiked at 20 µg/L.

3.2.3.4.3 Dilutions

Dilutions of samples depended on the type of sample delivered (FC, CK, PD, etc.). Initial analysis utilized different dilutions, as expected concentrations were not known

and concentrations varied by several order of magnitudes. Subsequently, similarity in order of magnitudes for certain types of sample allowed simultaneous analyses in two categories:

CK, CO, PD(X) : 1,000 fold dilution
F(X), LQ, RW, SR, PR, SW: 5,000 and 10,000 fold dilution.

The instrument detection limit (IDL) was calculated as the average concentration of a blank analyzed 10-12 times throughout an analysis day, plus three times the standard deviation. The IDL for Cs averaged 0.022 µg/L.

3.2.3.5 Inductively Coupled Plasma- Optical Emission Spectroscopy (ICPOES)

ICPOES was used by SREL to measure Na, Al, K, P, Ca, Mg, and Si. The Process Science Analytical Laboratory (PSAL) and AD laboratory were utilized in addition to SREL for analysis of some samples where additional verification was needed; these labs also analyzed for S by ICPOES.

3.2.3.5.1 Instrumentation

A Perkin-Elmer ICPOES Optima series 4300 DV was used providing the ability for simultaneous measurement in both an axial and radial view mode. A Perkin-Elmer AS91 autosampler was used for sample introduction.

3.2.3.5.2 Method

Yttrium at 2 mg/L was used as an Internal Standard to check for matrix effects on all samples, standards, and blanks. It was introduced online to the sample line using a spare channel on the peristaltic pump.

The method for sodium (Na) analysis included adding a Cesium ionization buffer at 1% w/v for all standards, samples and QC samples.

3.2.3.5.3 Dilutions

Dilutions of samples depended on the element and the type of sample delivered (FC, CK, PD, etc.). Initial analysis entailed running one element at a time because concentrations were not known and concentrations of specific element varied by several order of magnitudes. Subsequently, relative similarity in concentrations for some elements enabled them to be run simultaneously on one dilution. Elements were divided into 3 groups with dilutions depending on the sample type:

Na: 5000 and 10,000 fold dilution
Al and K: 10, 20, and 100 fold dilution.
Ca, Mg, Si, P: 5, 10, 20, and 100 fold dilution.

3.2.3.6 Ion Chromatography, Anions (ICA)

Ion Chromatography for anions (ICA) was used to measure oxalate, nitrate, nitrite, sulfate, phosphate, chloride, and fluoride. Only anions that were soluble in the sample or diluted sample were measured; any solids present were filtered out.

3.2.3.6.1 Instrumentation

A Dionex system fitted with a CD20 Conductivity Detector and a chromatography oven was used. The column used is a Dionex IONPAC® AS11-HC 4-mm analytical column preceded with an IONPAC® AG11-HC 2-mm guard column and an ATC-3 4-mm anion ion trap.

3.2.3.6.2 Method

Two methods were developed to account for samples matrix and interferences:

NO_2^- and NO_3^-:
Eluent: 10 mM NaOH
Eluent flow rate: 1.5 ml/min
Sample Volume: 10 µL

F^-, Cl^-, SO_4^{2-}, $C_2O_4^{2-}$, PO_4^{2-}
Eluent: 25 mM NaOH
Eluent Flow Rate: 1.5 ml/min
Sample Volume: 10 µL

3.2.3.6.3 Dilutions

Multiple dilutions of samples were necessary to account for differences in order of magnitudes in specific anions concentrations. Typical dilutions were dependent on the sample type:

For NO_2^- and NO_3^-: 100, 1000, 5000 and 10,000 fold dilution
For F^-, Cl^-, SO_4^{2-}, $C_2O_4^{2-}$, PO_4^{2-} : 25, 50, 100 and 1000 fold dilution

3.2.4 Samples Requiring Immediate Dilution

Samples requiring immediate dilution to prevent crystallization/precipitation of solids that may form upon cooling were taken from the sample point into a bottle containing sufficient deionized water to dissolve water soluble solids. The amount of deionized water used was typically about 10:1 by mass. The mass data were recorded so that the original concentration could be back-calculated. Because the dilutions performed were on a mass basis and the analyses of the diluted samples were reported on a mass per volume (mg/L) basis, the density of the original and diluted samples were also measured.

$$[X]_{original} = [X]_{diluted} \times \frac{\rho_{sample}}{W_{sample}} \times \frac{W_{water} + W_{sample}}{\rho_{diluted}}$$

3.2.5 Archive Samples

Numerous archive samples were taken throughout the tests. All samples analyzed also had a duplicate archive sample taken.

3.3 SRNL Work Instructions

The pilot scale testing was performed at the EDL. The testing was conducted following step by step instructions to operate various subsystems of the test facility. The Work Instructions were revised during testing as the test program progressed. The following is a list of all the revisions:

1. Fractional Crystallization Test Facility (U) Work Instruction, ITS-WI-0028 Rev. 0, 4-9-2008

2. Fractional Crystallization Test Facility (U) Work Instruction, ITS-WI-0028 Rev. 1, 4-14-2008

3. Fractional Crystallization Test Facility (U) Work Instruction, ITS-WI-0028 Rev. 2, 4-24-2008

4. Fractional Crystallization Test Facility (U) Work Instruction, ITS-WI-0028 Rev. 3, 5-5-2008

These work instructions are organized into sections that address how to run individual subsystems of the test facility. For each testing sequence, a detailed “run” plan identified the specific test objective, parameters of interest, acceptance criteria, and any special precautions to be observed.

The overall operation of the test facility for a particular test was achieved by executing the “run” plan following the instructions for individual subsystems.

While the detailed instructions are documented in above references, Figure 9 is an outline of instructions that provide an organization of instructions.

Figure 9. Work Instructions Outline

1.0 PURPOSE

2.0 SCOPE

3.0 Precautions/Limitations

- 3.1 Safety Considerations
- 3.2 PPE Requirements
- 3.3 Emergency Responses

4.0 Responsibilities

- 4.1 Acronyms
- 4.2 Responsibilities
  - 4.2.1 Overall Program Responsibilities
  - 4.2.2 SRNL Staff Responsibilities

5.0 Pilot Scale Test Facility Description

- 5.1 System Overview
- 5.2 Slurry Loop
- 5.3 Feed/Receipt Subsystem
- 5.4 Steam Supply
- 5.5 Condensate Subsystem
- 5.6 Centrifuge Subsystem
- 5.7 Product Dissolver Subsystem
- 5.8 Recycle Wash Subsystem
- 5.9 Spent Wash Subsystem
- 5.10 Cooling Water
- 5.11 Plant Air
- 5.12 Process Control

6.0 Data Acquisition

- 6.1 DAS
- 6.2 Note Book Records
- 6.3 Data Sheets/Rounds

7.0 Instructions for Fractional Crystallization Operations

- 7.1 Condensate System Startup and Shutdown
- 7.2 Operation of Steam Generator
- 7.3 Operation of Vacuum System
- 7.4 Material Transfer/Handling
  - 7.4.1 Filling Feed/Receipt Tanks from Simulant Totes
  - 7.4.2 Filling Crystallizer and Draw off Loop if they are at Atmospheric

8.0 Instructions for Fractional Crystallization Tests

8.1 Pre-Test Inspections
8.2 Post-Test Inspections
8.3 Test Run Plan
8.4 System Boilout with Water
8.5 Benchmark Chemical Tests
8.6 Baseline Chemical Tests
8.7 Feed Variability Tests

9.0 Attachments

9.1 Fractions Crystallization Pilot Scale Facility P&ID
9.2 Equipment List
9.3 Instrumentation List
9.4 Valve List
9.5 Fractional Crystallization Simulant Formula
9.6 Data Sheets/Rounds for Every Shift
9.7 Data Sheets/Rounds for Every Hour
9.8 Chemical Hazards Summary
9.9 Shift Turnover Form
9.10 Test Run Plan

3.4 Data Acquisition System

The DAS System was composed of National Instruments® (NI) Signal Processing and Acquisition hardware and NI Labview® DAS software coupled with a Dell Workstation computer.

3.4.1 DAS Hardware

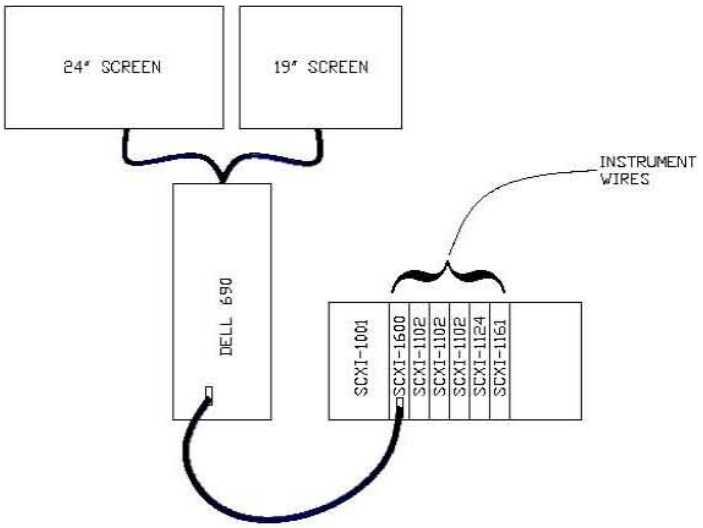
Computer:

Dell® Precision WorkStation 690
UltraSharp® 2408WFP 24-inch Widescreen Flat Panel Monitor with Height Adjustment Stand
UltraSharp® 1908FP 19-inch Flat Panel Monitor with Height Adjustable Stand

Signal Processing and Acquisition Hardware:

National Instruments®
SCXI-1001 12-Slot Chassis, U.S. 120 VAC
SCXI-1600, USB Data Acquisition and Control Module
SCXI-1102 32 ch Thermocouple Amplifier (3 Boards)
With SCXI-1303 32 ch Isothermal Terminal Blocks (3)
SCXI-1124 6 ch Isolated DAC Module
With SCXI-1325 Screw Terminal Block
SCXI-1161 8-Channel Power Relay Module

Figure 10. Wiring Schematic of DAS Hookup



EDL staff developed tables to correlate each instrument with a specific channel on the DAS (Tables 5 and Table 6).

3.4.2 DAS Software

EDL used National Instruments® Labview 8.2 Data Acquisition (and Control) Software for the DAS system. Each instrument was calibrated per the following SRNL procedures:

- ITS-0138, M&TE Calibration and Evaluation Process at Engineering Development Lab.
- Temperature Calibrations-ITS-0139, Comparative Temperature Calibrations at the Engineering Development Lab.
- Pressure Instrumentation-ITS-0140, Calibration of Pressure Measurement Devices Using a Mansfield and Green Pneumatic Weight Tester, Model RK.

Each instrument calibration resulted in mADC output versus sensed variable curve fit. Using this information and the calibration of the DAS system, a scale (transfer function) was developed for each instrument that provided an accurate readout in desired engineering units versus the read signal. These results are shown in Table 7.

Table 5. DAS Input Channel Allocation List for FC Instruments

DAS Chan.	Loop ID #	Instrument	M&TE #	Make	Model/Serial	Calibrated Range
0	CO01	Thermocouple	TR-03858	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
1	CW01	Thermocouple	TR-03857	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
2	CW03	Thermocouple	TR-03846	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
3	CO10	Thermocouple	TR-03908	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
4	CW06	Thermocouple	TR-03899	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
5	CW08	Thermocouple	TR-03901	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
6	CW10	Thermocouple	TR-03856	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
7	FR03	Thermocouple	TR-03851	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
8	PD01	Thermocouple	TR-03862	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
9	RW01	Thermocouple	TR-03906	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
10	RW05	Thermocouple	TR-03843	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
11	SL12	Thermocouple	TR-03845	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
12	ST02	Thermocouple	TR-03904	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-392 F
13	SW01	Thermocouple	TR-03852	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
14	SL04	Thermocouple	TR-03861	Omega	1/4", 12"L, Type E, Omega # GEQSS-14(G)-12	32-212 F
15	ST04	Thermocouple	TR-03905	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
16	ST05	Thermocouple	TR-03851	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
17	PD05	Thermocouple	03848 destrc	Omega	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	32-212 F
18	0	Thermocouple	0		0	0
19	0	Thermocouple	0		0	0
20	0	Thermocouple	0		0	0
21	0	Thermocouple	0		0	0
22	SL10	Transmitter	TR-03891	Omega	Omega CDTX-45T1 Conductivity analyzer	0-2 S/cm
23	PD06	Diff. Press Trans	TR-03490	Rosemount	Rosemount	0-100 in wc
24	~~SL05~~	~~Diff. Press Trans~~	~~TR-03493~~	~~Rosemount~~	~~Rosemount~~	~~0-100 in wc~~
25	SL06	Diff. Press Trans	TR-03691	Rosemount	Rosemount	0-200 in wc
26	SL08	Diff. Press Trans	TR-03106	Rosemount	Rosemount	0-30 in wc
27	SL02	Diff. T/couple	TR-03889	Omega	Omega 5TC-GG-E-20-72	0-20 C
28	ST01	DP across orifice	TR-03688	Rosemount	Rosemount	0-30 in wc
29	CO11	Magnetic FM	TR-03563		0	0-2 gpm
30	CO18	Magnetic FM	TR-03677		0	0-2 gpm
31	FR01	Magnetic FM	TR-03678		0	0-2 gpm
32	PD09	Magnetic FM	TR-03276		0	0-2 gpm
33	PD10	Magnetic FM	TR-03704		0	0-5 gpm
34	SW06	Magnetic FM	TR-03721		TR-03721	0-2 gpm
35	SW09	Magnetic FM	TR-03705		TR-03705	0-2 gpm
36	CO12	Press Transd	TR-03717			0-15 psia
37	SL07	Pressure Trans	TR-03885	Omega	Omega, PX4200-30VACI	0-15 psia
38	ST06	Pressure Transd	TR-03883	Omega	Omega, PX4200-30VACI	0-15 psia
39	PD07	Ultrasonic	N/A	Omega	Omega LVCN-51	0.3 - 6 ft
40	RW07	Ultrasonic	TR-03918	Omega	Omega LVU91	0.3 - 6 ft
41	SW07	0	N/A		Omega LVCN-51	0.3 - 6 ft
42	SL03	Ultrasonic FM	TR-03892	GE	GE TransPort PT878	0-1000 gpm
43	CO02	0	TR-03884	Omega	Omega CDCN-91AC	0
44	SL01	0	N/A	ABB/ITT	0	0-20 amps
45	SL01	0	N/A	ABB/ITT	0	0-3550 rpm
46	SL14	Magnetic FM	TR-03601		TR-03601	0-100 gpm
47	CW02	Magnetic FM	TR-03687		0	0-200 gpm
48	SL05	Diff. Press Trans	TR-03493	Rosemount	Rosemount	0-100 in wc
49	SL15	Ultrasonic FM	CDS-4714		Controllotron System 990	0-60 gpm
50	SL12	Mass Flow Meter	N/A	Brooks	Brooks flow controller Model # 58501A1BT342BEA	0-20 SLPM
51	0	0	0		0	0
52	0	0	0		0	0
53	0	0	0		0	0
54	0	0	0		0	0
55	0	0	0		0	0
56	0	0	0		0	0
57	0	0	0		0	0
58	0	0	0		0	0
59	0	0	0		0	0
60	PD05	Switch	0		0	On/Off
61	SW05	Switch	0		0	On/Off
62	PD07	Switch	0		0	On/Off
63	CO08	Switch	0		0	On/Off
64	CO33	Switch	0		0	On/Off
65	PD08	Switch	0		0	On/Off
66	RW05	Switch	0		0	On/Off
67	SW14	Switch	0		0	On/Off
68	RW06	Switch	0		0	On/Off
69	SW09	Switch	0		0	On/Off
70	SL06	Switch	0		0	On/Off
71	ALL LAH	Float Switches	0		0	On/Off

Table 6. DAS Output Channel Allocation for FC Instruments

Analog Out									
AO-0	AO-0	SV	CO32		Output to SV CO32	Valve Position Input	DAS	4-20 mA	MR
AO-1	AO-1	SV	SL12		Output to SV SL12	Valve Position Input	DAS	4-20 mA	MR
AO-2	AO-2	SV	FR05		Output to VFD for Pump FR Pump 1	Speed Control	DAS	4-20 mA	MR
AO-3	AO-3				Spare Analog Output Channel				
AO-4	AO-4				Spare Analog Output Channel				
AO-5	AO-5				Spare Analog Output Channel				
AO-6	AO-6				Spare Analog Output Channel				
AO-7	AO-7				Spare Analog Output Channel				
Digital Input/Output									
DIO-0	DIO-0				FR05 Power/Leeson VFD 174917	Power Supply Switch	DAS	NO Contact	MR
DIO-1	DIO-1				Spare Digital Input/Output Channel				
DIO-2	DIO-2				Spare Digital Input/Output Channel				
DIO-3	DIO-3				Spare Digital Input/Output Channel				
DIO-4	DIO-4				Spare Digital Input/Output Channel				
DIO-5	DIO-5				Spare Digital Input/Output Channel				
DIO-6	DIO-6				Spare Digital Input/Output Channel				
DIO-7	DIO-7				Spare Digital Input/Output Channel				

The DAS was calibrated per SRNL procedure L9.5-9133. Below are the results of each channel calibration.

Table 7. DAS Channel Calibrations

(Calibration check of all DAS channels was done on 1/30/2008)

	Thermocouples													
Temperature °C	Channel 0 T, applied	Channel 0 T, reading	Current °C	Channel 1 T, applied	Channel 1 T, reading	Temperature °C	Channel 2 T, applied	Channel 2 T, reading	Temperature °C	Channel 3 T, applied	Channel 3 T, reading	Temperature °C	Channel 4 T, applied	Channel 4 T, reading
0	0.0	0.1	0	0.0	0.1	0	0.0	0.4	0	0.0	0.3	0	0.0	0.2
25	25.0	25.1	25	25.0	25.1	25	25.0	25.3	25	25.0	25.2	25	25.0	25.2
50	50.0	50.2	50	50.0	50.1	50	50.0	50.3	50	50.0	50.2	50	50.0	50.2
75	75.0	75.2	75	75.0	75.1	75	75.0	75.3	75	75.0	75.2	75	75.0	75.2
100	100.0	100.2	100	100.0	100.1	100	100.0	100.3	100	100.0	100.2	100	100.0	100.2
125	125.0	125.2	125	125.0	125.1	125	125.0	125.3	125	125.0	125.2	125	125.0	125.2

Temperature °C	Channel 5 T, applied	Channel 5 T, reading	Temperature °C	Channel 6 T, applied	Channel 6 T, reading	Temperature °C	Channel 7 T, applied	Channel 7 T, reading	Temperature °C	Channel 8 T, applied	Channel 8 T, reading	Temperature °C	Channel 9 T, applied	Channel 9 T, reading
0	0.0	0.2	0	0.0	0.2	0	0.0	0.2	0	0.0	0.5	0	0.0	0.6
25	25.0	25.2	25	25.0	25.2	25	25.0	25.2	25	25.0	25.5	25	25.0	25.5
50	50.0	50.2	50	50.0	50.2	50	50.0	50.2	50	50.0	50.5	50	50.0	50.6
75	75.0	75.2	75	75.0	75.1	75	75.0	75.2	75	75.0	75.4	75	75.0	75.6
100	100.0	100.2	100	100.0	100.1	100	100.0	100.2	100	100.0	100.4	100	100.0	100.6
125	125.0	125.1	125	125.0	125.1	125	125.0	125.2	125	125.0	125.4	125	125.0	125.6

Temperature °C	Channel 10 T, applied	Channel 10 T, reading	Temperature °C	Channel 11 T, applied	Channel 11 T, reading	Temperature °C	Channel 12 T, applied	Channel 12 T, reading	Temperature °C	Channel 13 T, applied	Channel 13 T, reading	Temperature °C	Channel 14 T, applied	Channel 14 T, reading
0	0.0	0.6	0	0.0	0.7	0	0.0	1.0	0	0.0	1.0	0	0.0	1.0
25	25.0	25.5	25	25.0	25.7	25	25.0	26.0	25	25.0	26.0	25	25.0	25.9
50	50.0	50.5	50	50.0	50.7	50	50.0	50.9	50	50.0	50.9	50	50.0	50.8
75	75.0	75.5	75	75.0	75.6	75	75.0	75.9	75	75.0	75.9	75	75.0	75.8
100	100.0	100.5	100	100.0	100.6	100	100.0	100.9	100	100.0	100.9	100	100.0	100.8
125	125.0	125.5	125	125.0	125.6	125	125.0	125.9	125	125.0	125.8	125	125.0	125.8

Temperature °C	Channel 15 T, applied	Channel 15 T, reading	Temperature °C	Channel 16 T, applied	Channel 16 T, reading	Temperature °C	Channel 17 T, applied	Channel 17 T, reading	Temperature °C	Channel 18 T, applied	Channel 18 T, reading	Temperature °C	Channel 19 T, applied	Channel 19 T, reading
0	0.0	1.1	0	0.0	1.1	0	0.0	1.1	0	0.0		0	0.0	
25	25.0	26.0	25	25.0	26.1	25	25.0	26.1	25	25.0		25	25.0	
50	50.0	50.9	50	50.0	51.1	50	50.0	51.1	50	50.0		50	50.0	
75	75.0	75.9	75	75.0	76.0	75	75.0	75.9	75	75.0		75	75.0	
100	100.0	100.9	100	100.0	100.9	100	100.0	100.9	100	100.0		100	100.0	
125	125.0	125.9	125	125.0	125.9	125	125.0	125.9	125	125.0		125	125.0	

Temperature °C	Channel 20 T, applied	Channel 20 T, reading	Temperature °C	Channel 21 T, applied	Channel 21 T, reading	Current mA	Channel 22 V, meas.	Channel 22 V, calc.	Current mA	Channel 23 V, meas.	Channel 23 V, calc.	Current mA	Channel 24 V, meas.	Channel 24 V, calc.
0	0.0		0	0.0		4	2		4	2		4		
25	25.0		25	25.0		8	4		8	3.99		8		
50	50.0		50	50.0		12	6		12	5.99		12	BAD	
75	75.0		75	75.0		16	8		16	7.98		16		
100	100.0		100	100.0		20	10		20	9.98		20		

Current mA	Channel 25 V, meas.	Channel 25 V, calc.	Current mA	Channel 26 V, meas.	Channel 26 V, calc.	Voltage mVDC	Channel 27 T, meas.	Channel 27 T, calc.	Current mA	Channel 28 V, meas.	Channel 28 V, calc.	Current mA	Channel 29 V, meas.	Channel 29 V, calc.
4	1.99		4	2		0			4	2		4	1.99	
8	3.99		8	4.01		0.3			8	3.99		8	3.99	
12	5.98		12	6.01		0.6			12	5.99		12	5.98	
16	7.98		16	8.02		0.9			16	7.99		16	7.97	
20	9.97		20	10		1.3			20	9.98		20	9.96	

Current mA	Channel 30 V, meas.	Channel 30 V, calc.	Current mA	Channel 31 V, meas.	Channel 31 V, calc.	Current mA	Channel 32 V, meas.	Channel 32 V, calc.	Current mA	Channel 33 V, meas.	Channel 33 V, calc.	Current mA	Channel 34 V, meas.	Channel 34 V, calc.
4	1.99		4	2		4	2		4	2		4	2	
8	3.99		8	3.99		8	3.99		8	3.99		8	3.99	
12	5.98		12	5.99		12	5.99		12	5.99		12	5.99	
16	7.98		16	7.98		16	7.98		16	7.98		16	7.98	
20	9.97		20	9.98		20	9.98		20	9.97		20	9.97	

Current mA	Channel 35 V, meas.	Channel 35 V, calc.	Current mA	Channel 36 V, meas.	Channel 36 V, calc.	Current mA	Channel 37 V, meas.	Channel 37 V, calc.	Current mA	Channel 38 V, meas.	Channel 38 V, calc.	Current mA	Channel 39 V, meas.	Channel 39 V, calc.
4	2		4	2		4	2		4	2		4	2	
8	3.99		8	3.99		8	3.99		8	4		8	4	
12	5.99		12	5.99		12	5.99		12	6		12	6	
16	7.98		16	7.98		16	7.98		16	8		16	8	
20	9.98		20	9.98		20	9.97		20	10		20	9.99	

Current mA	Channel 40 V, meas.	Channel 40 V, calc.	Current mA	Channel 41 V, meas.	Channel 41 V, calc.	Current mA	Channel 42 V, meas.	Channel 42 V, calc.	Current mA	Channel 43 V, meas.	Channel 43 V, calc.	Current mA	Channel 44 V, meas.	Channel 44 V, calc.
4	2		4	1.99		4	2		4	2		4	2	
8	4.01		8	3.98		8	4		8	3.99		8	4	
12	6.01		12	5.98		12	6		12	5.98		12	6	
16	8.01		16	7.97		16	8		16	7.97		16	8	
20	10		20	9.96		20	10		20	9.96		20	10	

Current mA	Channel 45 V, meas.	Channel 45 V, calc.	Current mA	Channel 46 V, meas.	Channel 46 V, calc.	Current mA	Channel 47 V, meas.	Channel 47 V, calc.	Current mA	Channel 48 V, meas.	Channel 48 V, calc.	Current mA	Channel 49 V, meas.	Channel 49 V, calc.
4	2		4	2		4	2.01		4	1.99		4		
8	4		8	4		8	4.01		8	3.99		8		
12	6		12	5.99		12	6.02		12	5.98		12		
16	8		16	8		16	8.02		16	7.98		16		
20	10		20	9.99		20	10		20	9.97		20		

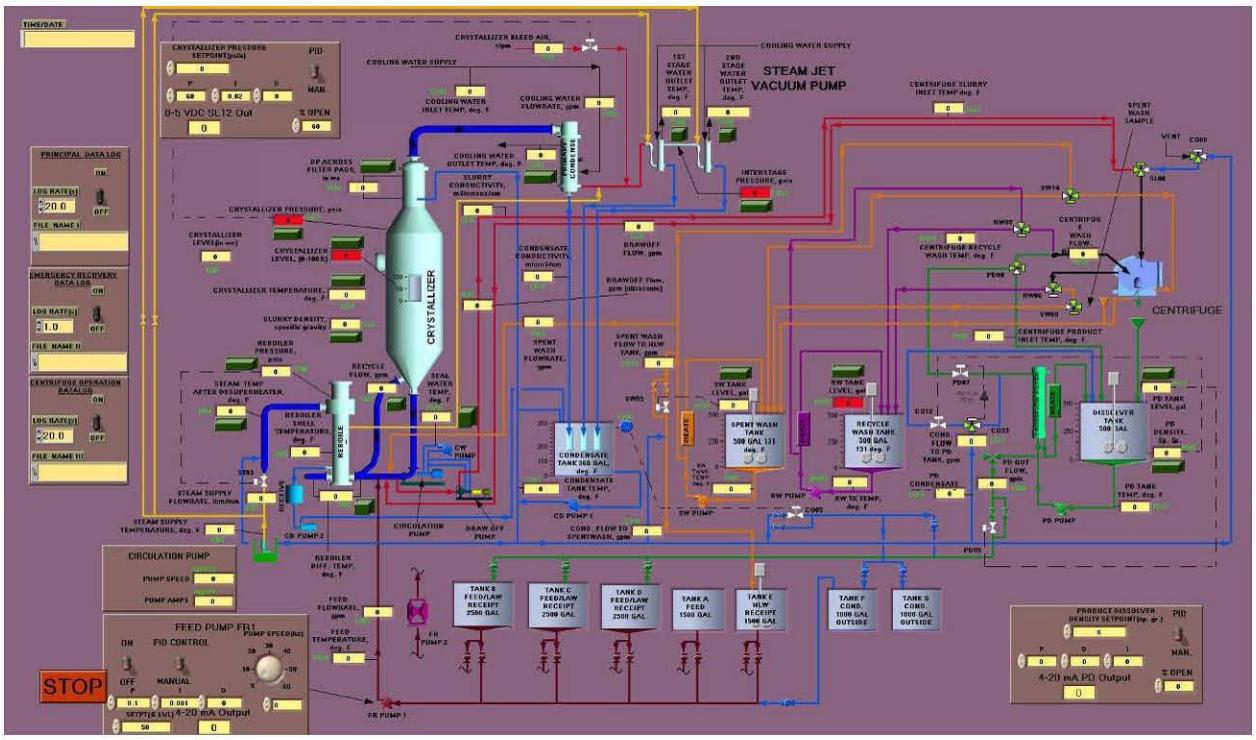
Table 8. Transfer Functions developed for each Channel on DAS

		Transfer Functions for DAS (Conversion from VDC to Engineered Units)			
DAS Channel	Instrument	Output	Slope (unit/VDC)		Intercept (Unit)
0	CO01	deg F			
1	CW01	deg F			
2	CW03	deg F			
3	CO10	deg F			
4	CW06	deg F			
5	CW08	deg F			
6	CW10	deg F			
7	FR03	deg F			
8	PD01	deg F			
9	RW01	deg F			
10	RW05	deg F			
11	SL12	deg F			
12	ST02	deg F			
13	SW01	deg F			
14	SL04	deg F			
15	ST04	deg F			
16	ST05	deg F			
17	PD05	deg F			
18	0				
19	0				
20	0				
21	0				
22	SL10	mS/cm	250.0000	x VDC +	-500.0000
23	PD06	in wc	0.5306	x VDC +	-0.0596
24	~~SL05~~	~~in wc~~	~~#DIV/0!~~	~~x VDC +~~	~~#DIV/0!~~
25	SL06	in wc	25.0501	x VDC +	-49.8247
26	SL08	in wc	3.7519	x VDC +	-7.5404
27	SL02	deg F	29.7652	x VDC +	-36.3335
28	ST01	in wc	3.7585	x VDC +	-7.5106
29	CO11	gpm	0.2542	x VDC +	-0.5046
30	CO18	gpm	0.2497	x VDC +	-0.4952
31	FR01	gpm	0.2506	x VDC +	-0.4989
32	PD09	gpm	0.2546	x VDC +	-0.5078
33	PD10	gpm	0.6483	x VDC +	-1.2963
34	SW06	gpm	0.2570	x VDC +	-0.5162
35	SW09	gpm	0.2515	x VDC +	-0.5035
36	CO12	psia	3.7587	x VDC +	-7.5060
37	SL07	psia	-1.8805	x VDC +	18.7472
38	ST06	psia	1.8699	x VDC +	-3.7499
39	PD07	gallons	76.7298	x VDC +	-240.3978
40	RW07	gallons	77.0141	x VDC +	-245.7047
41	SW07	gallons	78.2501	x VDC +	-258.8283
42	SL03	gpm	123.6250	x VDC +	-247.2500
43	CO02	µS/cm	125.6627	x VDC +	-255.9902
44	SL01	amps	2.1000	x VDC +	-4.2000
45	SL01	rpm	450.0000	x VDC +	-900.0000
46	SL14	gpm	12.6223	x VDC +	-25.0668
47	CW02	gpm	24.5897	x VDC +	-49.3739
48	SL05	Sp. Gr.	0.3913	x VDC +	0.2170
49	SL15	gpm	6.0069	x VDC +	0.0979
50	SL12	slpm	2.5000	x VDC +	-5.0000
51	0			x VDC +	
52	0			x VDC +	
53	0			x VDC +	
54	0			x VDC +	
55	0			x VDC +	
56	0			x VDC +	
57	0			x VDC +	
58	0			x VDC +	
59	0			x VDC +	
60	PD05	On/Off		x VDC +	
61	SW05	On/Off		x VDC +	
62	PD07	On/Off		x VDC +	
63	CO08	On/Off		x VDC +	
64	CO33	On/Off		x VDC +	
65	PD08	On/Off		x VDC +	
66	RW05	On/Off		x VDC +	
67	SW14	On/Off		x VDC +	
68	RW06	On/Off		x VDC +	
69	SW09	On/Off		x VDC +	
70	SL06	On/Off		x VDC +	
71	ALL LAH	On/Off		x VDC +	

3.4.3 DAS Pictures

Figures 11 and 12 are examples of the DAS screens available to operating staff.

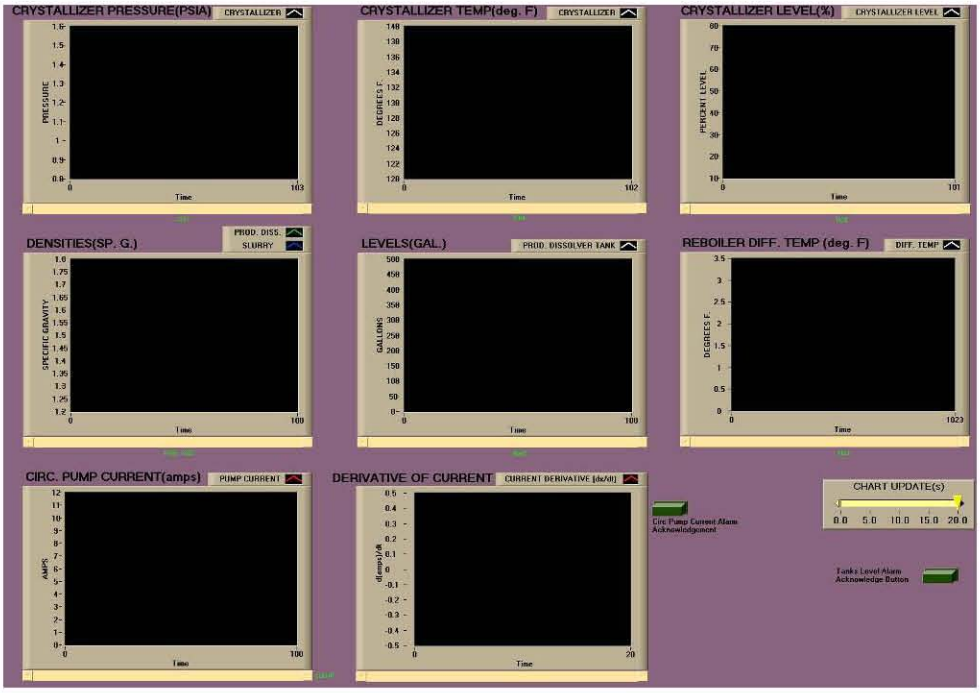
Figure 11. Main DAS Screen



The main screen displayed digital values of all DAS instrumentation. Solenoid valve positions were also indicated by color change (green open, white closed). Tank levels in the PD, RW, and SW tanks were also displayed as an analog fill line. Additional displays on the main DAS screen included control panels for the FR Pump 1 (main simulant feed pump), SL12 (bleed air upstream of steam jet pumps, and PD tank density (controlled through valve CO32 condensate addition to PD Tank). Three dialog boxes provided file name and location, frequency (seconds between logging), and status of three separate data logs.

In addition to the main DAS screen, a trending screen was provided to display critical parameter changes historically. This was useful in trending system operations. Figure 12 shows a trending screen without the charted values displayed.

Figure 12. Trend Chart Screen on Additional Monitor for the FC Test Rig DAS.



Additionally, Figures 13A through 13E show the hardware installation.

Figure 13. DAS Hardware Installation.

Figure 13A

Figure 13B

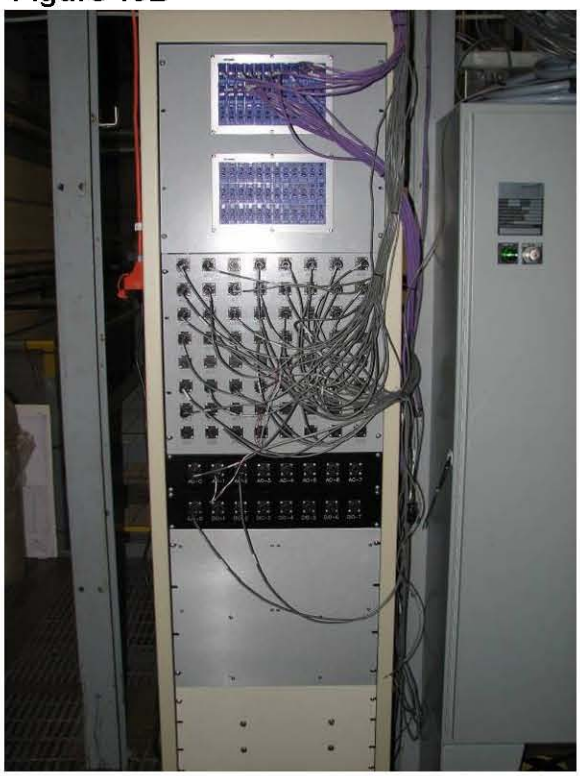
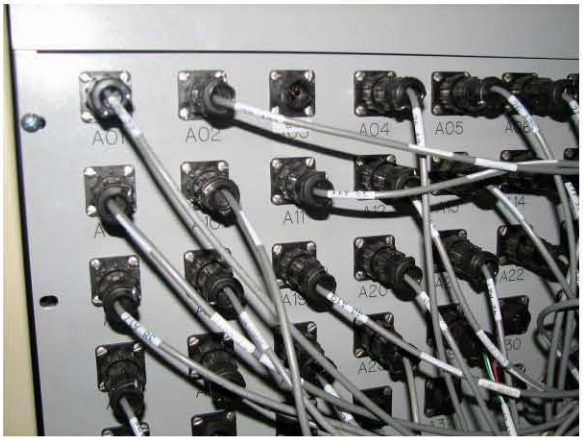


Figure 13C

Figure 13D

Figure 13E



3.4.4 DAS References

- P&ID, latest version
- SRNL Procedure L9.5-9133
- ITS-0138, M&TE Calibration and Evaluation Process at Engineering Development Lab
- Temperature Calibrations-ITS-0139, Comparative Temperature Calibrations at the Engineering Development Lab
- Pressure Instrumentation-ITS-0140, Calibration of Pressure Measurement Devices Using a Mansfield and Green Pneumatic Weight Tester, Model RK.

3.5 FCPP Training

Training the EDL engineers and operators was an important part of the FCPP test planning because the process fundamentals and some of the pilot equipment, e.g. centrifuge and crystallizer, were new to the existing staff. A two-phase approach

was adopted. The first phase was vendor training for the EDL engineers followed by vendor presentations to the operators. The second phase was called "in-house" training conducted by EDL staff and the training was focused on the implementing procedures the operators would use during the tests.

3.5.1 Vendor Training

Initial vendor training for EDL staff was conducted December 11 and 12, 2007 by Swenson. The focus of the training was the theory and operation of the crystallizer system and theory and operation of the centrifuge. The Swenson draft "Operating Instructions" for the crystallizer was reviewed with the staff because they would become the basis for EDL "Work Instructions" used for day-to-day operations. Following classroom training staff assembled in the EDL to become familiar with the actual hardware during installation. The second vendor training was conducted February 26, 27, and 28, 2008 and included Swenson along with two Field Engineers from Krauss Maffei Process Technology (KMPT) who discussed the theory and operation of the centrifuge. During this time the centrifuge was operated for the first time (using only water) to check machine functions and load the latest software modifications in the centrifuge PLC.

3.5.2 In-House Training

In addition to the vendor training described above, EDL performed in-house training based upon the vendor training material as well as Work Instructions written specifically to operate the Pilot-Scale Test Facility. The training was conducted by the PI and the Lead Designer of the Test Facility. The following staff received this training:

1. Four Test Engineers
2. Four Laboratory Technicians
3. Four other Support Engineers

The training was performed in two phases:

1. Classroom training based upon the Work Instructions and Vendor Material.
2. Practical Factors: This included hands-on training on the centrifuge and a detailed facility walkdown.

All the training material was prepared and documented in accordance with the guidelines provided by the SRNL Training Group.

4.0 TEST EXECUTION AND RESULTS

The testing period was initiated April 17, 2008 when initial benchmark tests began and was concluded on May 31, 2008 upon completion of the baseline testing. Benchmark testing was performed from April 17 through May 20, 2008. Baseline testing was begun on May 20 immediately after cesium nitrate addition to the FC feed stream and continued through May 31, 2008.

The baseline objectives of a cesium DF of at least 50 and the sodium reporting to the product stream were attained. Actual operations during benchmark testing produced an average product cake cesium DF of 130 and an average product dissolver cesium DF of 58. The difference between the cesium DF determined in the product dissolver vs. the filter cake was the result of cesium contaminated liquor overflow from the centrifuge to the product dissolver. The estimated sodium yield based on overall mass balances during the period May 28 through May 31 was approximately 52%.

Due to operational and instrumentation difficulties, it was necessary to operate the process in a manual mode throughout the test period. Manual operation of crystallizer pressure and level resulted in large fluctuations in crystallizer temperature and slurry density which reduced the efficiency of the centrifuge operation and reduced throughput of the system. In addition, the failure of the cross-flow filter in the product dissolver circuit allowed fines to be recycled to the centrifuge, further reducing centrifuge capacity.

While manual operations stretched the test engineer's capabilities and response times, the overall system functioned acceptably with some periods of stable operations.

4.1 Benchmark Testing

Benchmark testing consisted of adjusting parameters to produce crystals that allowed for easy dewatering and efficient washing of the centrifuge cake, adjusting centrifuge parameters to consistently deliquor and wash process crystals, and perform shutdown tests to demonstrate the ability to shutdown or idle the system and return the system to normal operations.

Acceptance criteria for successfully completing Benchmark Testing included:

- The system should operate smoothly to produce crystals that allow for easy dewatering (deliquoring) and efficient washing of the centrifuge cake.
- From previous laboratory simulant work, the dominant crystal (sodium nitrate) should have a CSD mode size in the range of 300 to 450 microns.
- The centrifuge should cycle (spin, wash, & discharge) smoothly in an automatic mode while producing an easily removable cake.
- The crystallizer system should shutdown safely to a condition that facilitates restart of the system.

The pilot system was mainly operated in manual control because of instrumentation problems. Even so, operators were able to control the most important system parameters, i.e. temperature and pressure in the crystallizer and various system flows and tank levels. The burkeite and sodium carbonate crystals remained small, as observed in previous laboratory testing, and were slow to dissolve in the Product Dissolver Tank. Slow dissolution had been evaluated by the earlier laboratory work so the EDL staff had designed a cross-flow filter system to remove the "fines" (generally considered to be crystals in the 10 micron range). However the unit soon became unusable due to low permeability and was removed from service. The slurry CSD was checked on a regular basis and confirmed a primary mode size in the range of 300 to 450 microns. Problems in the centrifuge feed system (from the slurry draw-off loop) resulted in erratic centrifuge operation. However once the system was reconfigured the centrifuge was able to operate in an automatic mode to control the feed, spin, wash, and discharge cycles. As noted above operational problems caused unplanned shutdowns, but the system was easily restarted.

Due to operational difficulties, primarily related to the centrifuge production capacity, pilot feed rates were limited to one-half or less of the design rate. When operating at low feed rates, other plant parameters were adjusted accordingly. The reduced pilot feed rates were used throughout the remainder of testing and are presented in Table 9.

4.1.1 Crystallizer Loop

The crystallizer loop performed as anticipated and crystals formed as predicted, i.e., burkeite (a sodium double salt containing sulfate and carbonate) formed first followed by sodium carbonate monohydrate, followed by sodium nitrate and then other sodium salts. Due to the non-functionality of the cross-flow filter in the Product Dissolver Tank, the small crystals were continually recycled to the centrifuge and crystallizer and impacted the overall crystal size distribution in the crystallizer and, may have impacted centrifuge solids separation and decontamination capability.

Based on earlier model projections and experiments, the key operating conditions and parameters for the crystallizer loop were identified in the test plan and are included in Table 10 below.

Table 9. Reduced Pilot Feed Rates

	Control Valve	Flowmeter	Flowsheet based on expected centrifuge performance	Flowsheet based on actual centrifuge performance	Notes
Centrifuge Cycle, sec			240	961	
Number of cycles			50	8	
Total cleanout time, min			2	30	
Wash 1 instantaneous flow			5.00	3.50	
Wash 2 instantaneous flow			5.00	3.10	
Cake/cycle (lb)			27	29.6	1
Cake (lb/min)			6.61	1.50	
Crystallizer Feed Rate			1.50	0.34	
Wash 1 duration/cycle, sec			23	36	
Wash 1 average flow			0.47	0.11	
Wash 2 duration/cycle, sec			22	40	
Wash 2 average flow			0.46	0.10	
Condensate to PD Tank	CO32	CO11	0.56	0.13	2
PD Product (average)			0.53	0.12	
40% of PD Product	PD04	PD09	0.21	0.05	3
80% of PD Product	PD06	PD09	0.42	0.10	3
Condensate added to make LAW			0.42	0.10	
40% of cond added to LAW	CO03	CO24	0.17	0.04	4
80% of cond added to LAW	CO04	CO24	0.34	0.08	4
SW recycle to crystallizer	SW07	SW09	1.30	0.29	5
SW Product (average)			0.29	0.07	
Flow through valve SW04	SW04	SW06	0.12	0.03	6
Flow through valve SW06	SW06	SW06	0.23	0.05	6
Condensate to make HLW	CO31	CO18	0.29	0.07	7
Steam (lb/min)	ST03	ST01	11.0	2.5	

Notes:

1 This is determined based on the peel start height averaged over all the cycles between heel removals.

2 This flow should be controlled to maintain the PD tank specific gravity at 1.44. Unfortunately, valve CO32 leaks through about 0.12 gpm, so it will be necessary to close valve CO35 if the flow needs to be stopped completely.

3 At very low flow rates there is not much point in trying to set the flow through valve PD06. Simply set the flow through valve PD04 to about 0.30 gpm, and let the PD tank level controller stop and start flow as necessary to maintain level in the tank. Unfortunately, the level control tends to give false highs when the level is either too high or too low, so it may be necessary to manually shut off the flow to prevent draining the tank.

4 At very low flow rates there is not much point in trying to set the flow through valve CO04. Simply set the flow through valve CO03 to about 0.30 gpm, and let the Condensate tank level controller stop and start flow as necessary to maintain level in the tank.

5 Unfortunately, at very low flows the recycle line tends to plug, so it may be necessary to flush it occasionally with a high flow (or condensate) and then reset the desired flow.

6 At very low flow rates there is not much point in trying to set the flow through valve SW06. Simply set the flow through valve SW04 to about 0.30 gpm, and let the SW tank level controller stop and start flow as necessary to maintain level in the tank. Unfortunately, the level control tends to give false highs when the level is either too high or too low, so it may be necessary to manually shut off the flow to prevent draining the tank.

7 The centrifuge cleanout procedure adds more than enough water into the SW tank, so flow through valve CO31 is not actually needed.

Table 10. Test Plan Initial Key Operating Conditions/Parameters

Parameter	Units	Minimum	Nominal	Maximum
Recirc Pump Flow Rate (SL03)	gpm	550	600	650
Recirc Pump Speed (SL01)	rpm	TBD	1907	TBD
Feed Flow Rate (FR01)	gpm	0.5	1.5	2.0
Reboiler Shell Side Steam Pressure (ST06)	psia	2	12.8	14.7
Reboiler Inlet Steam Temp (ST04)	°F	50	96	100
Slurry Reboiler Delta T (SL02)	°F	0	2	3
Crystallizer Level (SL06)	%	20	50	80
Crystallizer Pressure (SL07)	psia	0.97	1.35	1.74
Slurry Density (SL05)	SpG	1	1.6	2
Slurry Temp in Crystallizer (SL04)	°F	131	140	149

Benchmark testing demonstrated that the recirculation pump flow rate and speed were controllable, even operating the equipment in the manual mode. The same was true for the slurry reboiler inlet steam temperature control and control of the differential temperature across the slurry reboiler. However, due to the non-steady state of the overall FC system, control of the feed flow rate, crystallizer level and pressure, and the slurry level within the crystallizer required constant attention to maintain process control. The causes for non-steady state operations include lower than planned centrifuge feed rates, plugged lines, varying crystal size distribution causing centrifuge solids separation difficulties, instrumentation difficulties, and test engineers undergoing the learning curve.

The following graphs demonstrate the variability in parameter conditions during normal operations of the crystallizer loop during the later stages (May 16 beginning at 1409 hours through May 18 at 1055 hours) of benchmark testing when operations were considered more stable, but not steady state.

Figure 14. Graph of Crystallizer Temperature and Pressure

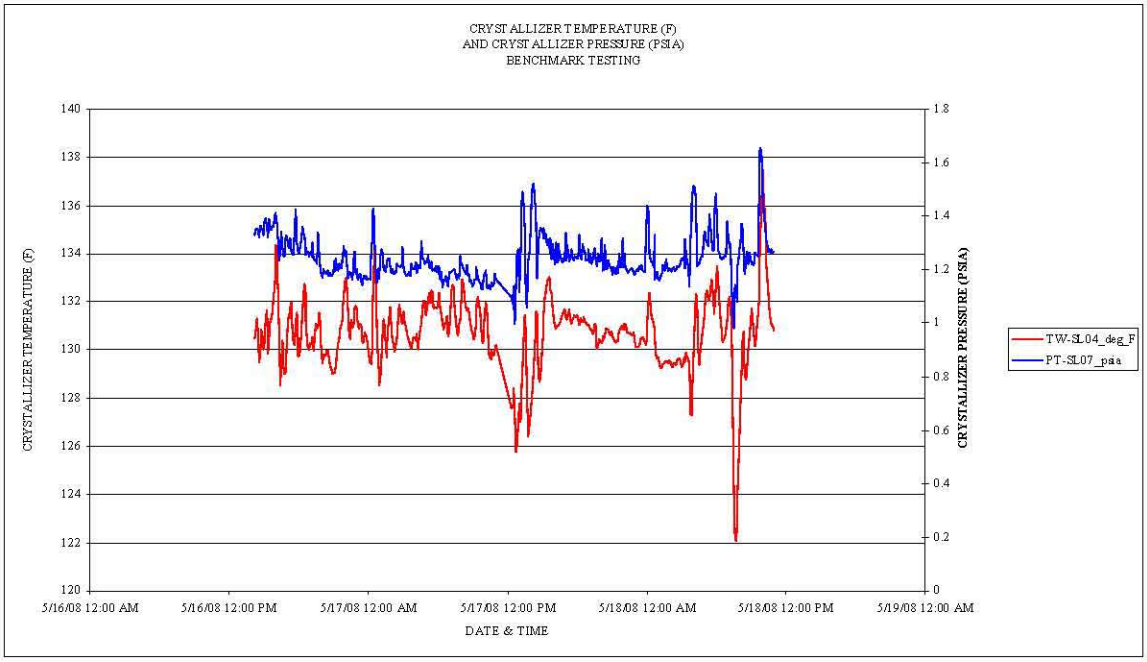


Figure 14 indicates that small fluctuations in manual pressure control (±0.25 psia) resulted in large changes in crystallizer temperature (±10ºF) due to raising and lowering the boiling point pressure and temperature. A sudden drop in boiling point temperature causes a drop in solubility of sodium nitrate resulting in the nucleation of fine particles. Fine sodium nitrate crystals reduce the efficiency of the centrifuge by creating a low permeability filter cake.

Figure 15. Graph of Reboiler Steam Temperature, Delta T and Pressure

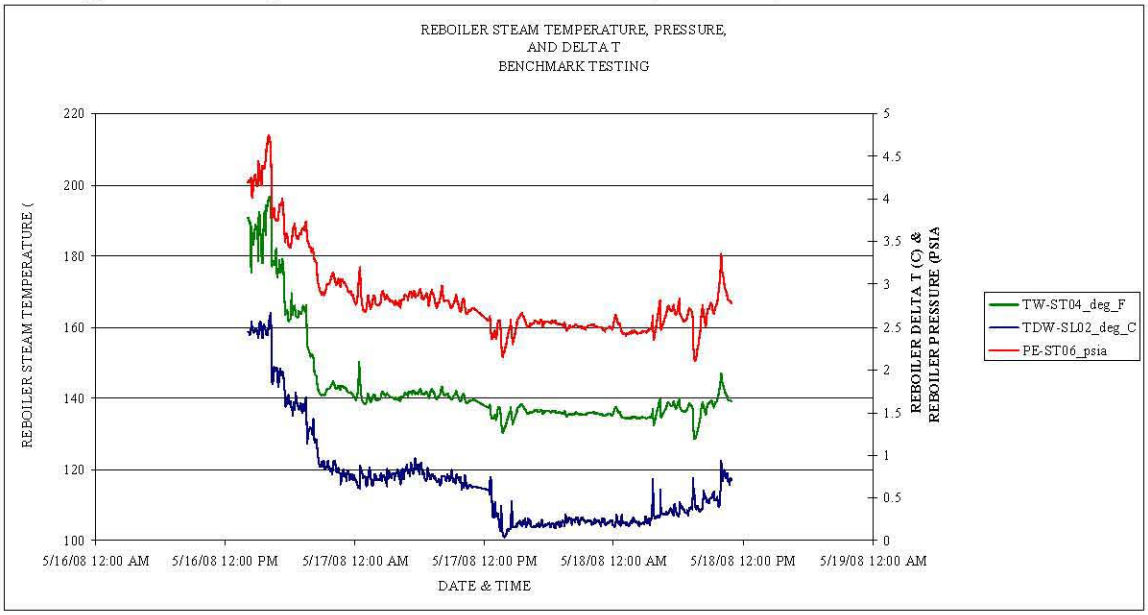


Figure 15 indicates that reboiler steam temperature and pressure and the process heating rate (ΔT) responded normally. The steam condensed at its saturation temperature at the shell operating pressure; heat transferred to the process in direct proportion to the temperature difference between the steam and the process.

Figure 16. Graph of Crystallizer Level and Slurry Density

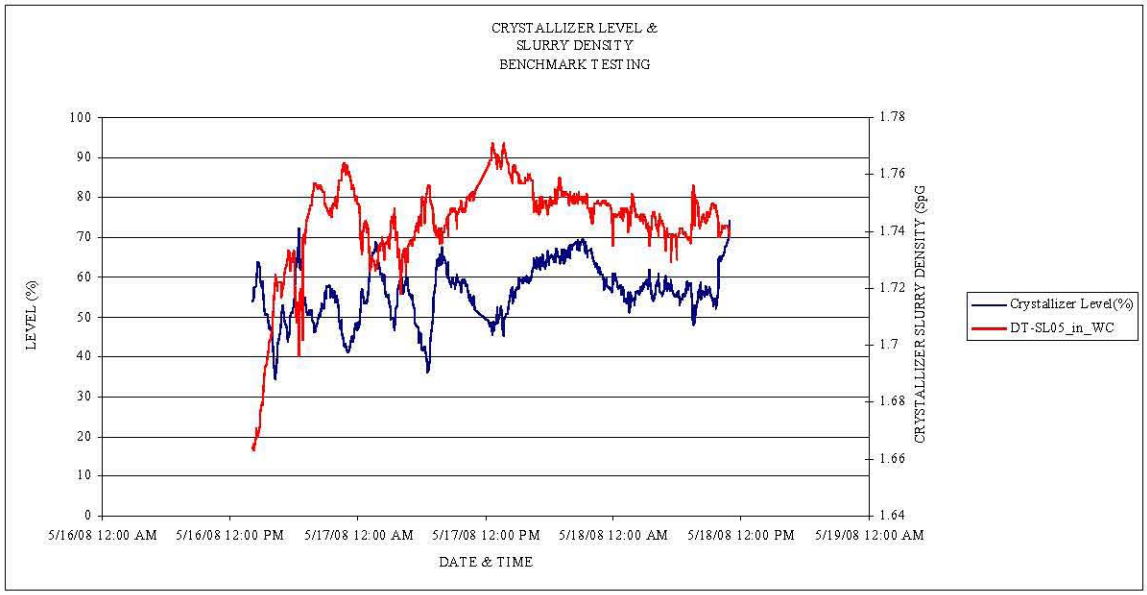
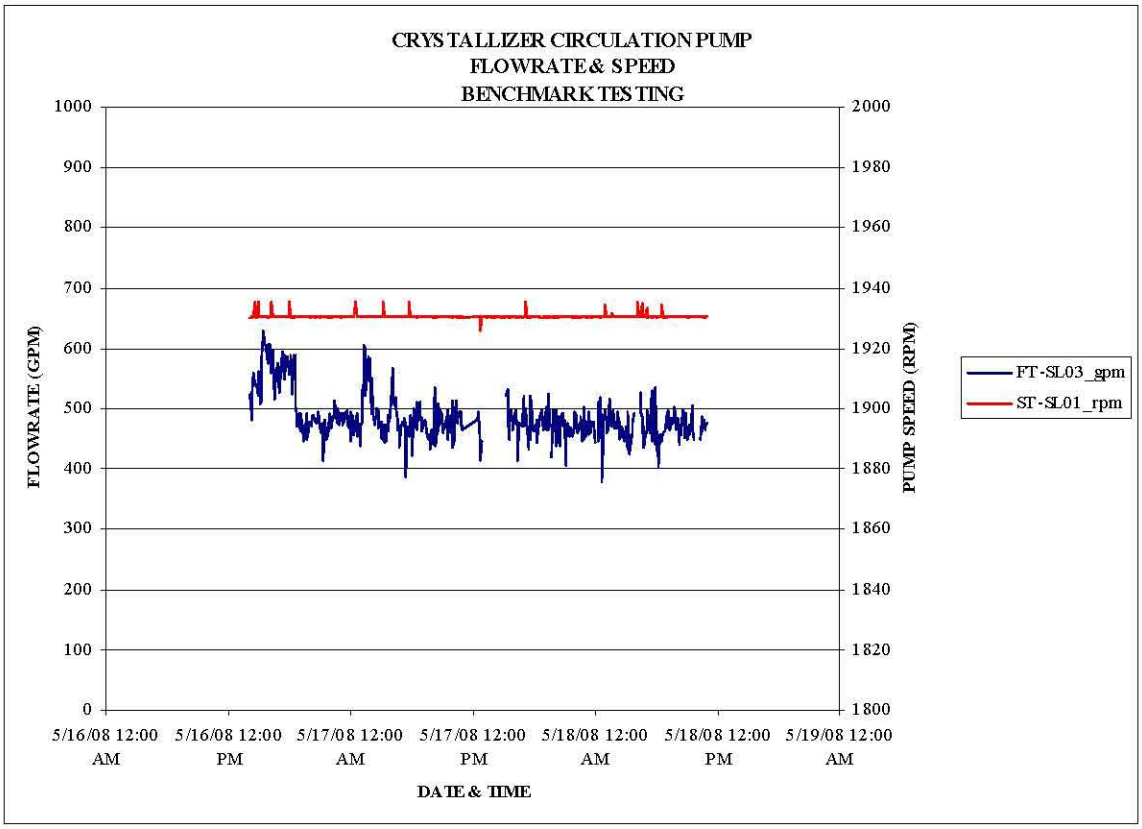


Figure 16 indicates that the crystallizer density had an opposite response to the crystallizer level. When the level was low, slurry density was high. When the level was high, slurry density was low. This effect indicates an imbalance between the feed and the evaporation rates caused by manual control of the crystallizer level and reboiler steaming rate.

Although slurry density varied by only 3.5% (1-1.76/1.70 = 3.5%) during this period, measurements of undissolved solids (solids/total mass) varied by ~10% (50 $\pm$ 5%). This indicates that small variations in slurry density (caused by dilution, evaporation, and/or temperature swings) caused large variations in the crystal mass in the crystallizer. Rapid changes in crystal mass results in poor crystal size distribution and morphology and poor solid/liquid separation.

Figure 17. Graph of Crystallizer Circulation Pump Flowrate and Speed



The crystallizer circulation pump flowrate and speed were set at a constant flowrate of 600 gpm and 1907 rpm per the test plan. Figure 17 indicates a relatively constant actual flow rate of about 500 gpm. The pump speed was consistently about 1930 rpm. This indicates that the fluid in the crystallizer was pumpable and didn't exert undo stress on the pump. It is unknown why the flowrate was lower and speed was higher but both are within the operating limits for the pump.

4.1.2 Centrifuge Loop

During the early stages of benchmark testing, numerous problems occurred in the centrifuge loop. Those problems included feed line blockages, overfeeding or slugging the centrifuge such that feed materials overflowed the centrifuge into the discharge lines during centrifuge operations, difficulties with salt cake removal and centrifuge plugging. Numerous changes were made to the centrifuge loop to correct these problems; restrictor valves and line flushes were installed and tested to reduce pluggage of the feed line and allow the proper feed volume to be charged to the centrifuge such that slugging of the centrifuge was minimized or eliminated, proper cleaning cycles and frequencies were determined to assure adequate heel removal and more automatic mode operations, the salt cake discharge chute was modified to include a Teflon liner to enhance the dry solids discharge from the centrifuge during the peel step and better attention to the slurry feed density was identified as a requirement to provide a uniform feed stream to the centrifuge.

Again choosing the later stages of the benchmark testing (May 16 to May 18) as the period for final benchmark testing, most if not all of the problems encountered early in the testing had been resolved such that extended centrifuge loop operations could occur. During the final testing the operating conditions were generally stable with some intermittent process upsets and recovery periods. The slurry feed density variability and the amount of fines in the feed resulting from the failure of the cross-flow filters impacted extended centrifuge operations. During the final benchmark testing the centrifuge was operated for between 9 and 12 batches before heel removal was required. Before cake removal, each batch received 2 wash cycles of between 6 and 10 seconds each. During the testing period the centrifuge loop received only one emergency shutdown and this was due to the peel knife being stuck at the 2 mm peel position which caused excessive vibration and automatic shutdown.

Figure 18. Product Dissolver Density

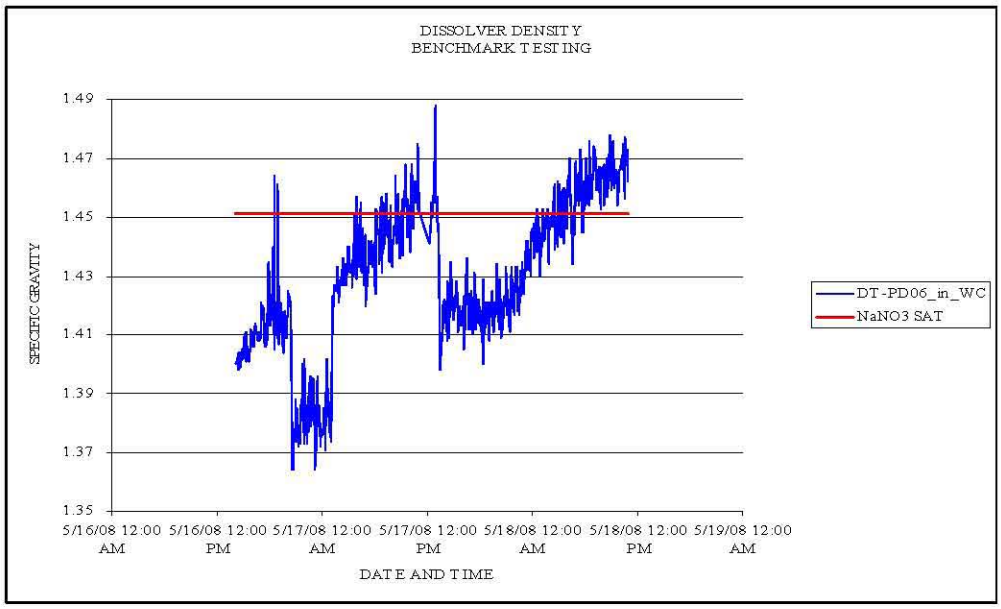


Figure 18 shows a wide variation in product dissolver density during benchmark testing because of manual control of the dissolution water feed rate. The set point for product dissolver density was 1.45, the saturation point for sodium nitrate at 131ºF. At a density of 1.45, the wash solution recycled to the centrifuge does not dissolve sodium nitrate in the centrifuge cake. At lower density, the wash solution partially dissolves the centrifuge cake thereby reducing yield, crystal size distribution, cake permeability, and the extent of deliquoring.

Centrifuge cake permeability was further reduced by the recycle of entrained fines from the dissolver product because of the failure of the cross-flow filter. At a density of 1.45, sodium sulfate and carbonate are supersaturated and are entrained in the centrifuge wash solution. The recycled fines reduce cake permeability and the extent of cake deliquoring.

4.1.3 Support Systems

Support systems to the FC Pilot Plant included SRNL provided HVAC, condenser cooling water and plant air. No proven problems were identified with these systems. It was thought that the cooling water flow rates decreased about 0300 and 1500 every day causing pilot process swings but investigations provided no data to support this theory.

4.1.4 Sample Drawing and Analysis

Changes and additions made to the analytical plan have been previously described in Section 3.2. In this section, the general frequencies of samples and analyses are described for the Benchmark test.

From startup of the crystallizer on 4/16/08 at 1200 hours (hrs) until the occurrence of crystal nucleation, slurry recirculation (SR) samples were taken periodically and analyzed for density, total solids (TS), and undissolved solids (UDS). Nucleation of sodium carbonate monohydrate and burkeite occurred much more quickly than anticipated (4/16/08 1800 hrs), so the sampling frequency was not switched to that specified for the time period from nucleation until 30 wt% UDS was reached. Due to confusion about the definition of nucleation, this switch was not made until 4/18/08 at 1700 hrs. Nucleation had been assumed to mean nucleation of the sodium nitrate, which had not yet occurred. It was decided that nucleation would mean any crystal species that had nucleated. On 4/16/08 at 2000 hrs, the UDS in the crystallizer was about 4 wt%.

At 4/18/08 1700 hrs, the SR samples began to be analyzed by TEASV at a 2 hr frequency. TS and UDS were also analyzed at a 2 hr frequency. Occasional PLM samples were also taken. At this time the UDS in the crystallizer was about 11 wt%. At about 2100 hrs on 4/18/08, the UDS jumped from about 14 to 36 wt% and remained at 36-45 wt% until the system was shut down on 4/19/08 at about 1700 hrs.

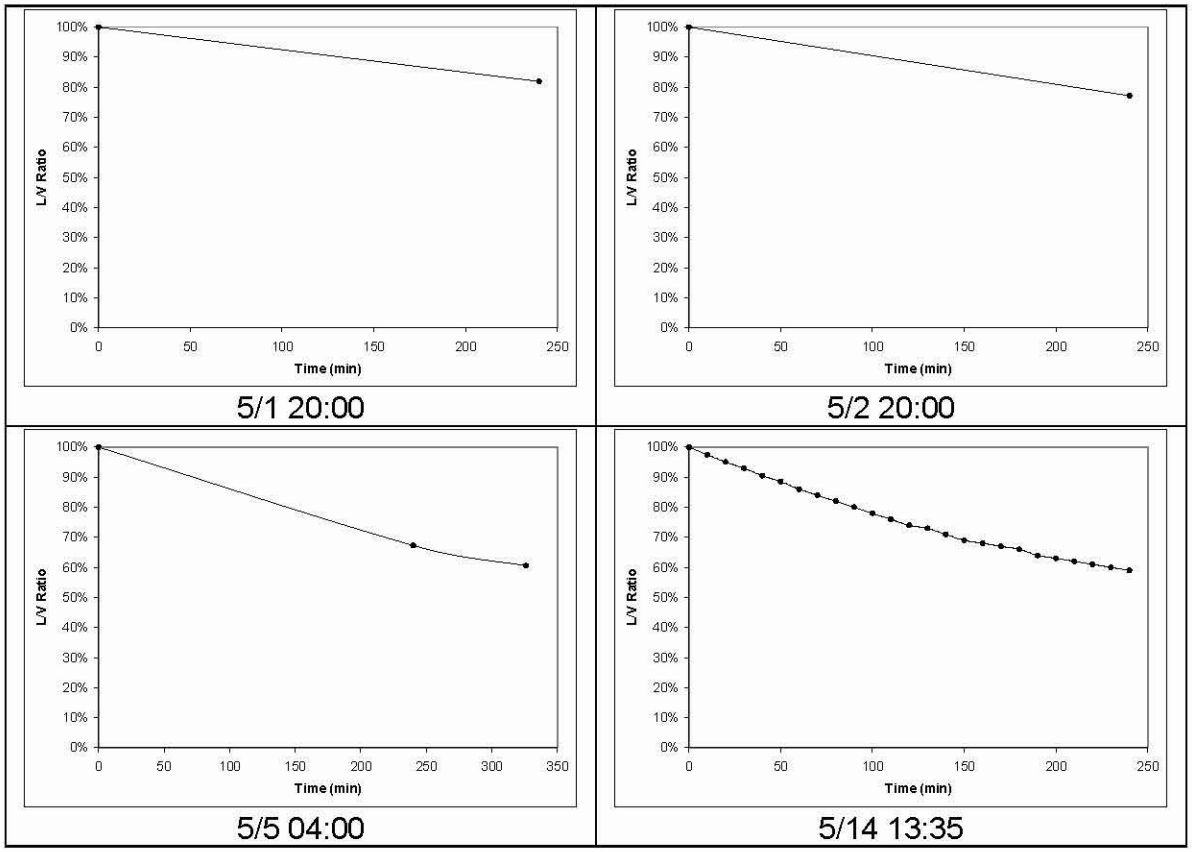
The FC system was restarted on 4/25/08 at 1200 hrs. Sampling SR began and samples were analyzed for density, TS, and UDS. On 4/26/08 at 0800 hrs TEASV and TS/UDS samples were taken at 2 hour intervals and CSD samples were taken at 4-8 hour intervals.. On 4/26/08 at 0800 hrs the UDS in the crystallizer was about 20 wt%. The CSD frequency was switched to 8 hours on 4/27/08 at 0800 hrs.

On 5/1/08 at 1200 hrs, regular SW density samples, occasional cake samples, and a daily PD archive sample were started. On 5/7/08at 2100 hrs, the first daily RW density sample was taken. These samples were continued until 5/10/08 at 0600 hrs when a pump seal failure forced the system to be shut down.

Several typical TEASV plots are shown in Figure 19. Note that the data for 5/5/08 go to about 330 minutes versus 240 minutes for the other plots. For most plots made, only the last data point was graphed because the data was very close to linear over the 4 hour interval. These data do show that the settling rate did increase from

5/2/08 to 5/4/08 and that after restart, on 5/14/08 the settling rate was about the same as it was on 5/5/08.

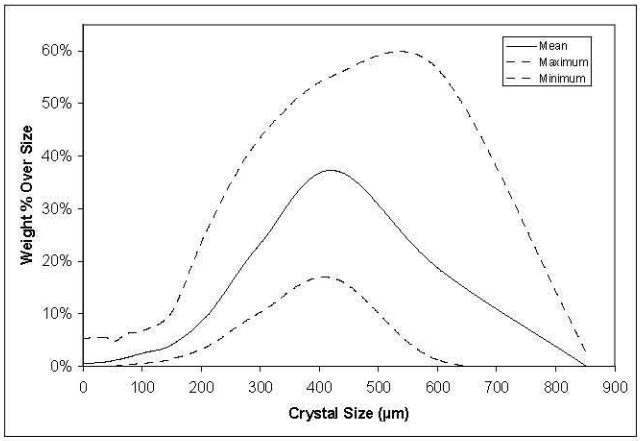
Figure 19. Typical TEASV Plots

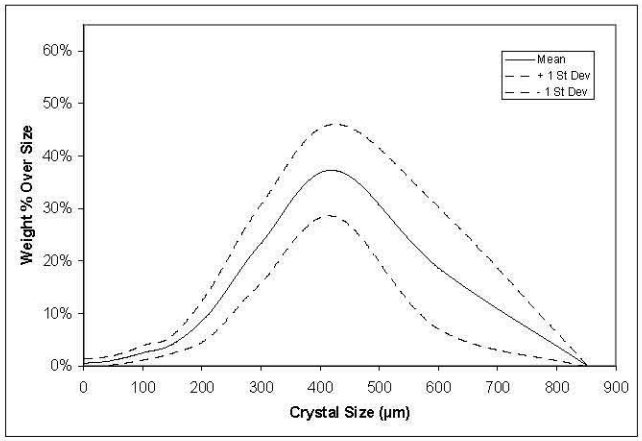


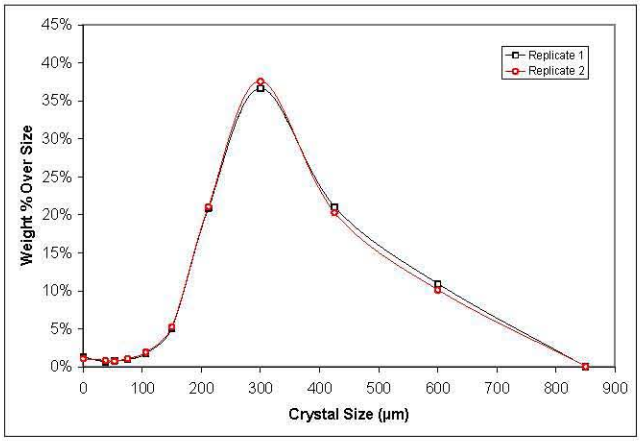
The system was again restarted on 5/16/08 at 1900 hrs and SR sampling and analysis for density and TS/UDS began at 2100 hrs. On 5/17/08 at 1400 hrs, regular 4 hour SW samples for density were begun. The SR samples were analyzed for TEASV for 4 hour duration at 4 hour intervals, density was measured every 2 hours, CSD samples taken every 8 hours, PLM samples every 2-4 hours, and TS/UDS and Quick UDS samples every 4 hours. RW samples were taken once per day and analyzed for density. These sampling frequencies were maintained until the Baseline test began on 5/20/08.

CSD was performed on 74 washed, dried slurry recirculation (SR) samples and two CK samples. Of the SR samples, 25 were taken during the baseline testing and 49 during the benchmark testing. The data shown below are for the entire test, both benchmark and baseline. The mean CSD of all the SR samples is shown in Figure 20 along with the maximum and minimum percentages at each sieve size and the average ± 1 standard deviation. Typical CSD plots for maxima at 300, 425, and 600 μm are also shown. The CSD maximum location for each sample is shown in the last graph. A value of 513 was plotted for samples where the wt% at 425 and 600 μm were approximately equal.

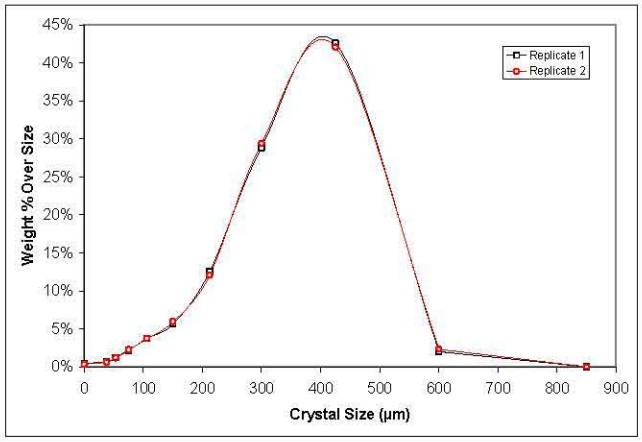
Figure 20. Average CSD for Slurry Samples



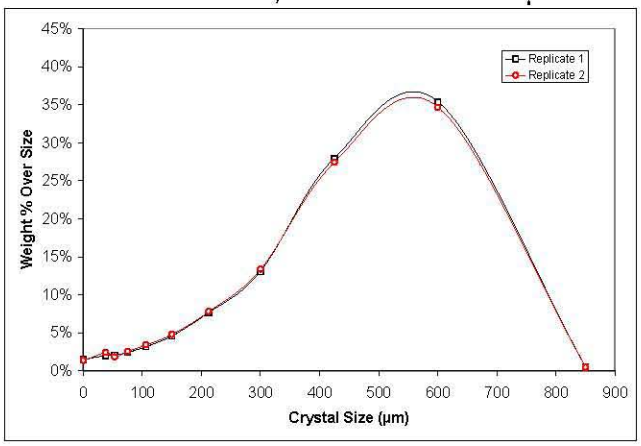




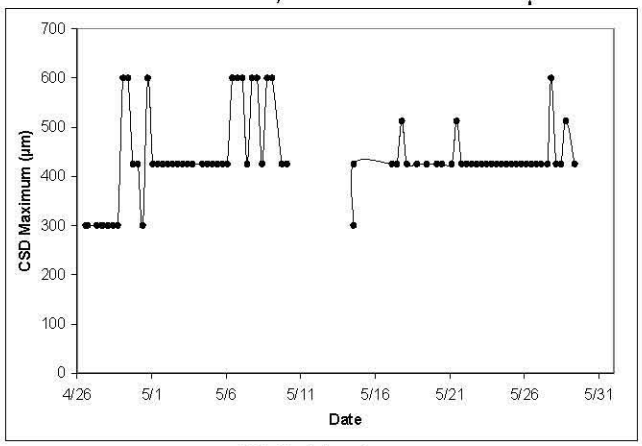
SR-0428-1000; maximum at 300 µm



SR-0505-1000; maximum at 425 µm



SR-0509-0200; maximum at 600 µm



CSD Maxima

4.1.5 Off-Normal Operations

During the pilot testing period a variety of equipment and operational problems allowed a full suite of casualty recoveries to be implemented that had originally been planned as drills for testing mode 5. The following actual casualties occurred during this period of testing:

- Loss of steam.
- Loss of feed and condensate.
- Loss of instrumentation (Data Acquisition System – DAS).
- Pipe break and pipe plugging.
- Loss of power.
- Trip of the centrifuge.
- Loss of crystallizer control (temperature, level, and pressure).
- Loss of process tank pumps (spent wash, recycle, & product dissolver).
- Loss of process tank recirculation line heaters. The process tank heaters were installed with fast burn fuses. Replacement with slow burn fuses appeared to solve the problem

4.2 Baseline Testing

Baseline Testing established a continuous mode of system operation using the full SST Early Feed simulant, except for chromium which was omitted for continued safety concerns. The main objective was to establish that the pilot system could meet the product sodium yield and Cs decontamination predictions/expectations. In support of the IPS Project, an additional objective was to determine the ability of the system to separate sulfate from the LAW stream and produce a high sulfate co-product stream. The acceptance criteria for successfully completing Baseline Testing were that the simulated LAW product had to meet or exceed:

- Cs DF of at least 50.
- Na fraction in LAW product is equal to or greater than 50% of the Na content in the feed stream as determined by laboratory analysis.

Baseline testing began on May 20th 2008 after the addition of Cs and continued until process shutdown on May 31st 2008.

Due to the limited baseline testing window it was agreed that no testing time was available to evaluate IPS Project requirements.

As during benchmark testing the baseline operating condition was operated in manual control. Pilot feed rates were the same as those developed and used during the benchmark testing. All centrifuge improvements (i.e., Teflon sleeve for the discharge chute, wash rates and frequencies, charge rates, etc) continued to be used for baseline testing. Overall, the FC Pilot system was easily and consistently controlled. The test engineers and on-shift staff seemed much more comfortable with the operation of the system.

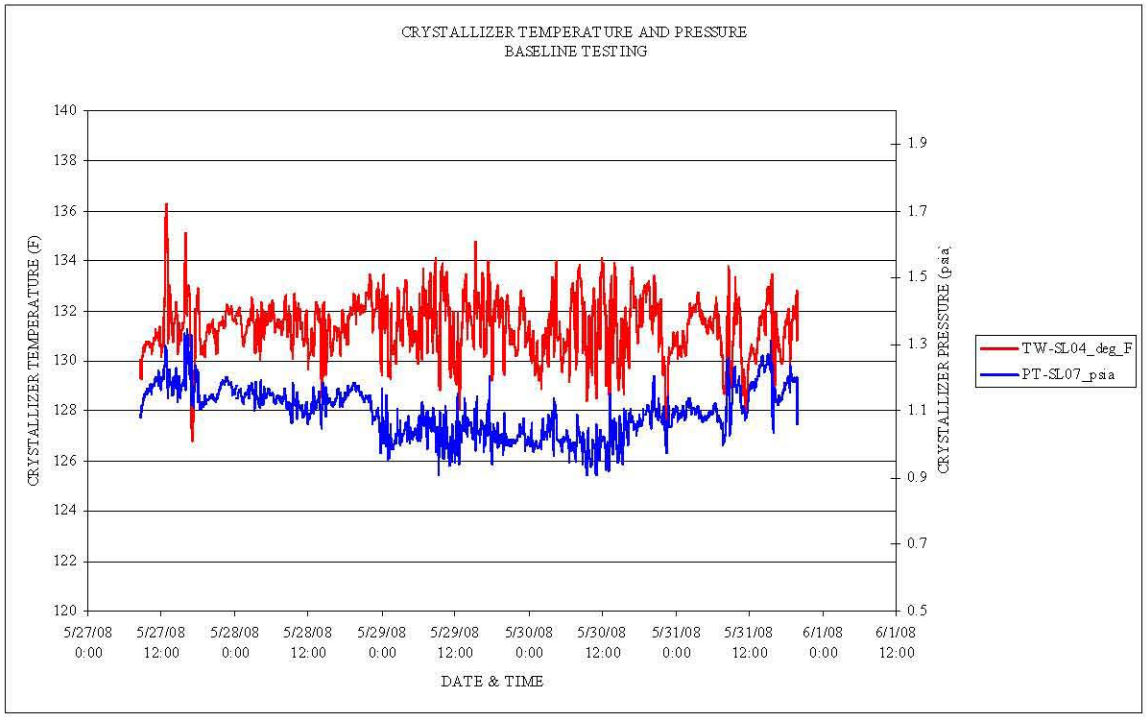
4.2.1 Crystallizer Loop

The crystallizer loop continued to perform as planned and utilized the same operating parameters and conditions as utilized during benchmark testing.

Baseline testing demonstrated that the recirculation pump flow rate and speed was controllable, even operating the equipment in the manual mode. The same was true for the slurry reboiler inlet steam temperature control and control of the differential temperature across the slurry reboiler. However, due to the non-steady state of the overall FC system, control of the feed flow rate, crystallizer level and pressure, and the slurry level within the crystallizer required constant attention to maintain process control. The causes for non-steady state operations include lower than planned centrifuge feed rates, plugged lines, varying crystal size distribution causing centrifuge solids separation difficulties, instrumentation difficulties. It deserves note here that operator experience and improved response to process upsets resulted in improved process control.

The following graphs demonstrate the variability in parameter conditions during normal operations of the crystallizer loop the baseline testing activities.

Figure 21. Graph of Crystallizer Temperature and Pressure



During baseline testing, crystallizer pressure and temperature fluctuated due to manual control of the crystallizer vacuum. Small pressure fluctuations cause changes in boiling point temperature and solubility of sodium nitrate. A sudden drop in boiling point temperature causes a drop in solubility of sodium nitrate resulting in the nucleation of fine particles. Increases in boiling point temperature and pressure increase the solubility of sodium nitrate and result in dissolution of fine crystals and edge rounding of large crystals.

Figure 22 indicates that reboiler steam temperature and pressure and the process heating rate (ΔT) responded normally during baseline testing. The steam condensed at its saturation temperature at the shell operating pressure; heat transferred to the process in direct proportion to the temperature difference between the steam and the process.

Figure 22. Graph of Reboiler Temperature, Pressure, and Delta T.

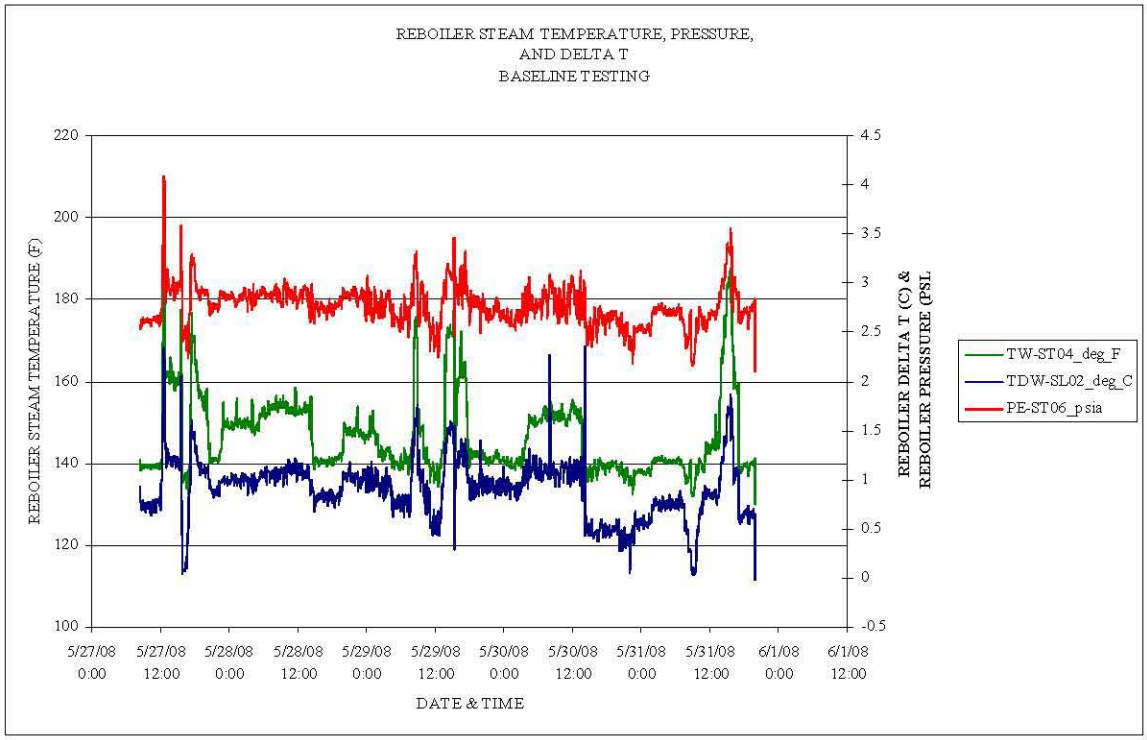


Figure 23. Graph of Crystallizer Level and Slurry Density.

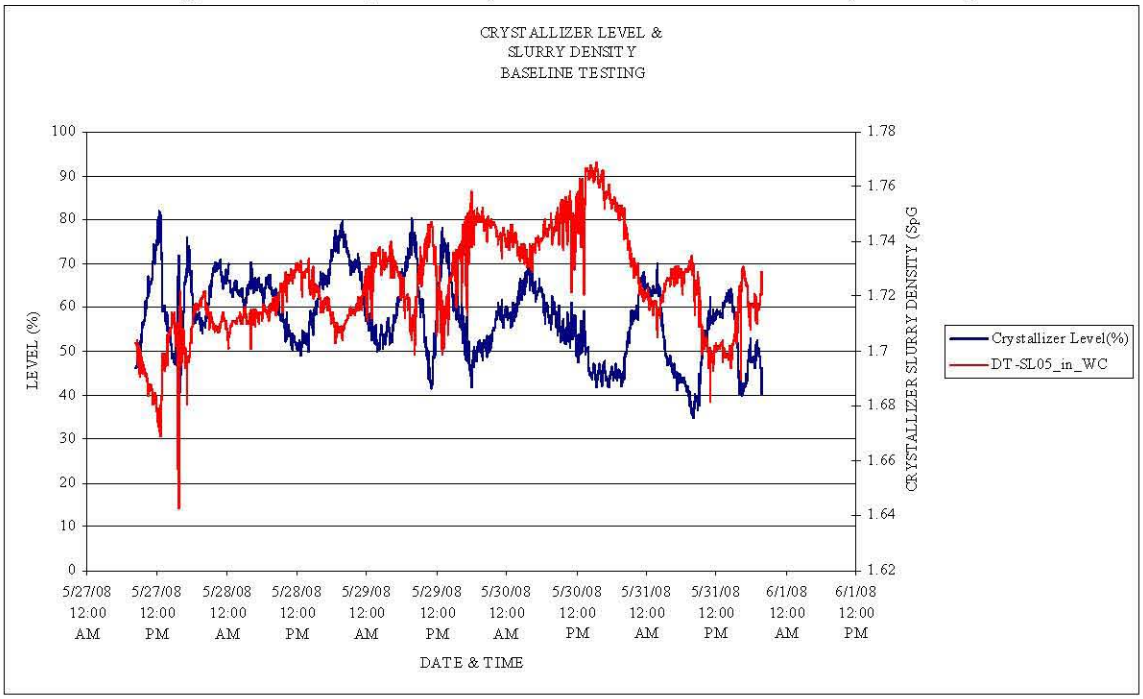


Figure 23 indicates that the crystallizer density had an opposite response to the crystallizer level. When the level was low, slurry density was high. When the level was high, slurry density was low. This effect indicates an imbalance between the feed and the evaporation rates caused by manual control of the crystallizer level and reboiler steaming rate.

Although slurry density varied by only 5.0% (1-1.68/1.70 = 5.0%) during this period, measurements of undissolved solids (solids/total mass) varied by ~10% (50 $\pm$ 5% - see Figure 23). This indicates that small variations in slurry density (caused by dilution, evaporation, and/or temperature swings) caused large variations in the crystal mass in the crystallizer. Rapid changes in crystal mass results in poor crystal size distribution and morphology and poor solid/liquid separation.

It is important to note that only limited data were available for the crystallizer circulation pump flow rate and speed during the baseline testing. It is assumed that the crystallizer circulation pump continued to perform as indicated earlier during benchmark testing. Checks of the operating logs and alarms indicated no problems with the system.

4.2.2 Centrifuge Loop

During the final testing the operating conditions were generally stable with some intermittent process upsets and recovery periods. The slurry feed density variability and the amount of fines in the feed resulting from the failure of the cross-flow filters impacted extended centrifuge operations. During the final baseline testing the centrifuge was operated for between 9 and 12 batches before heel removal was required. During baseline testing it was determined that the number of washes and duration of the wash had an impact on the Cs decontamination factor achieved. Centrifuge Cs DF is shown in Figure 24 for the period 5/21-5/28/2008. The average centrifuge cake Cs DF was 130. The large variability in DFs observed in Figure 24 were the result of varying cake washing time and volume of wash solution. The large DFs (100 to 300) shown during 5/21/08 and 5/22/08 were the result of following the flowsheet values for wash time and wash volume for the cake before cake removal. The reduced DFs (50 to170) obtained from 5/23/08 until the end of baseline testing were the result of reducing the wash times and wash volumes to ¼ and ½ of the flowsheet values.

Figure 24. Centrifuge Cake Cs DF

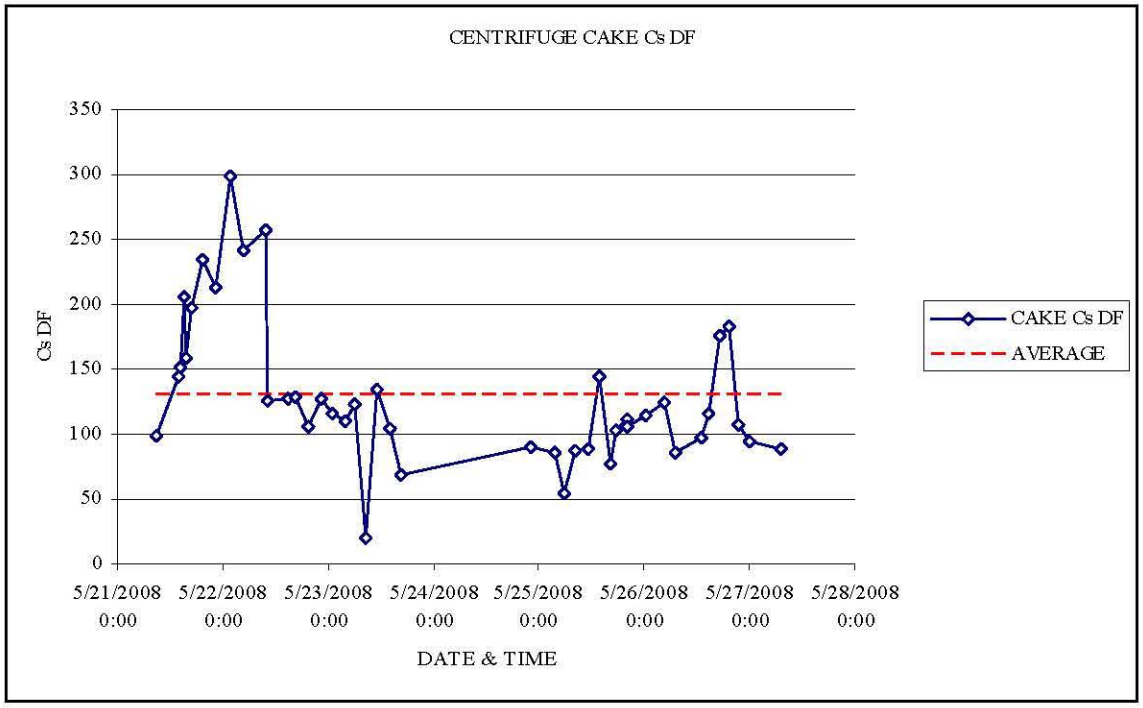
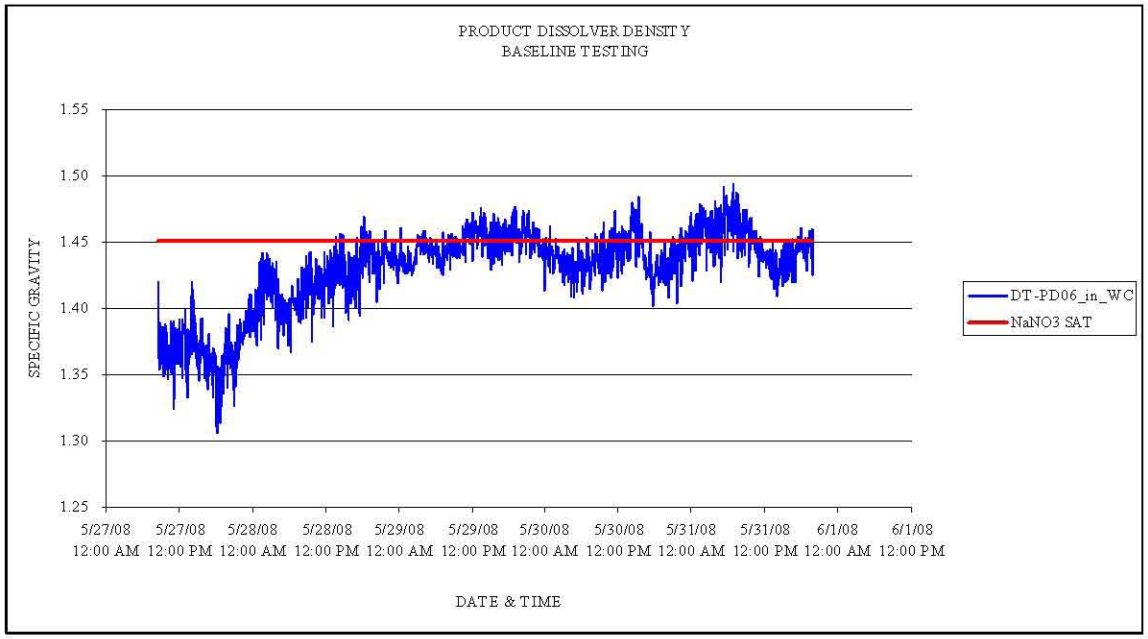


Figure 25 shows that during baseline testing the dissolver slurry density was manually controlled closer to sodium nitrate saturation, thus reducing the amount of dissolution of the centrifuge cake. However, the extent of cake decontamination may have been reduced by the recycle of entrained fines from the dissolver product because of the failure of the cross-flow filter. At a density of 1.45, sodium sulfate and carbonate are supersaturated and are entrained in the centrifuge wash solution. The recycled fines reduced cake permeability and the extent of cake decontamination.

Figure 25. Product Dissolver Slurry Density.



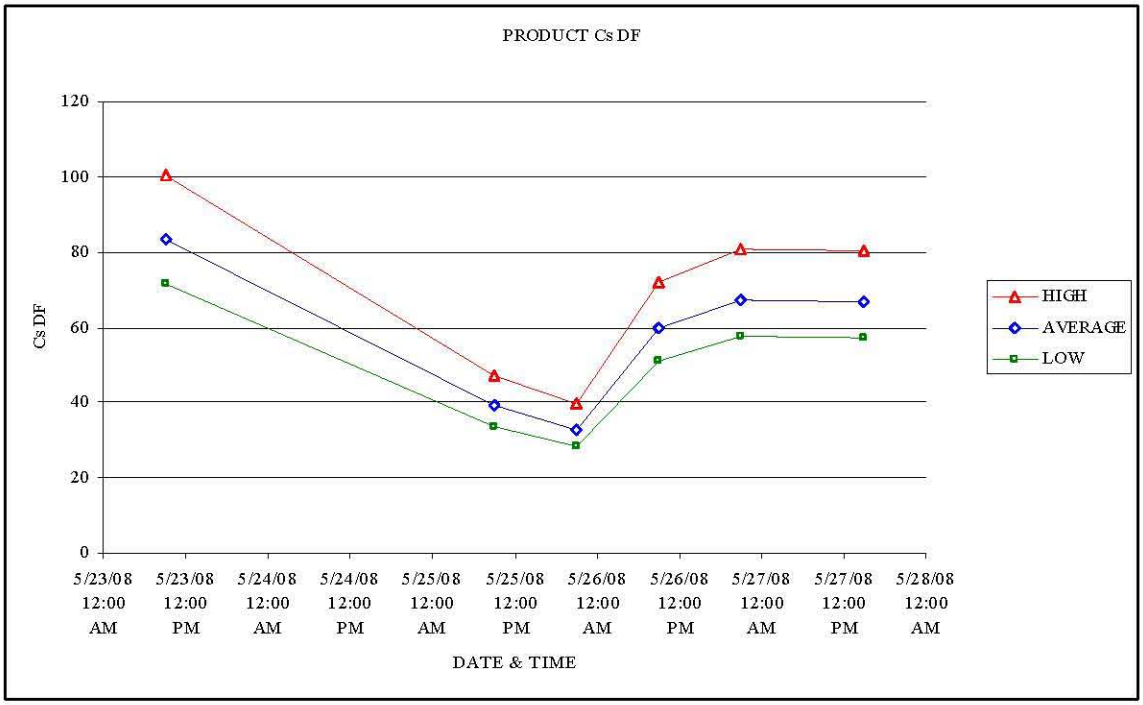
Cesium DF measured in the product dissolver averaged 58 for the period 5/23-5/28/2008. Product Cs DF is calculated as:

$$\text{Cs DF} = (\text{Na/Cs})_{\text{PRODUCT}}/(\text{Na/Cs})_{\text{FEED}}$$

Product dissolver Cs DF values are shown in Figure 26. However, since only two values of feed (Na/Cs) were measured (488 & 347), the uncertainty in this calculation is ±15%, which is shown as the high and low points on the graph.

The product Cs DF is substantially lower than product cake DF measurements (58 vs. 130) and results achieved in laboratory tests. This may have been due to occasional cesium contaminated liquor overflow from the centrifuge to the product dissolver.

Figure 26. Product Dissolver Cs DF.



4.2.3 Support Systems

Support systems to the FC Pilot Plant included SRNL provided HVAC, condenser cooling water and plant air. No proven problems were identified with these systems.

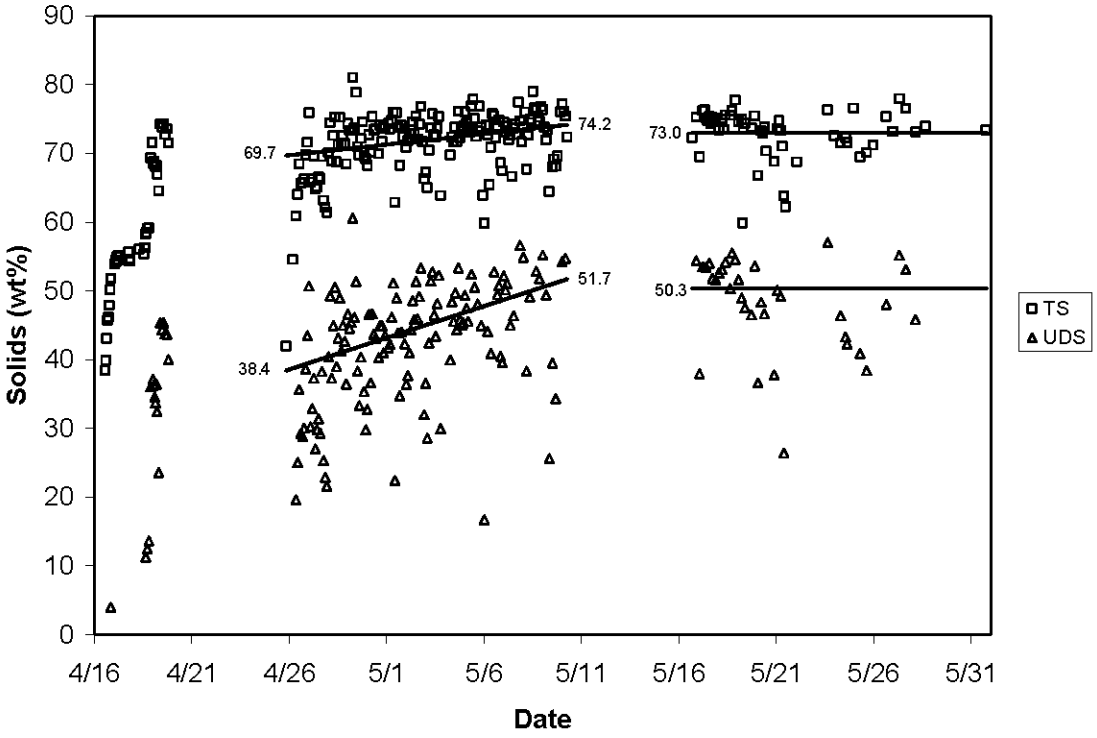
4.2.4 Sample Drawing and Analysis

On 5/20/08, the Cs was added to the feed and the crystallizer. On 5/21/08 at 0200 hrs, cake samples at about 4 hour intervals were taken for archive; the SR and SW samples remained the same, while RW samples were now taken every 4 hours. At 1000 hrs, archive samples of the HLW receipt tank E (PR) began to be taken at 4 hour intervals, and one sample per day was analyzed for elements and Cs by ICPOES and ICPMS. At 1700 hrs, the SR sampling load was reduced to PLM samples every 4 hours and CSD samples every 8 hours plus two samples per day for elements and Cs. CK samples from every 2nd peel began to be analyzed for elements and Cs. At 1300 hrs a complete analysis of Feed Tanks A and B were performed; analyses included elements, Cs, anions, carbonate, and free OH.

4.2.5 Results

Graphs of the TS, UDS, supernate solids (SS), and densities for both the benchmark and baseline tests are shown in Figures 27-29. Figure 27 shows that the TS and UDS both increased during the benchmark test (the data were fit linearly with selected outliers removed); there are less data for the baseline test, but the average values shown are very close to the last fitted values from the benchmark test.

Figure 27. Total Solids and Undissolved Solids in Slurry Recirculation Samples.



The supernate solids (SS), which are the total solids in the supernate decanted at 131°F from the ASV test, are shown along with the TS in Figure 28. The SS values decreased slightly after the start of the tests and reached a fairly constant value of about 49 wt%. Figure 29 shows the UDS measured from the TS and SS compared with the Quick UDS (Q-UDS) analysis results. As expected, the Quick UDS values were higher than the UDS values because of the residual supernate liquid in the filtered solids. On average, the Q UDS values were about 10-15 wt% higher than the UDS values (e.g., 60 wt% vs. 45-50 wt%). Or, in other words, The Q-UDS values were about 133% of the UDS values.

Figure 28. Total and Supernate Solids in Slurry Recirculation Samples

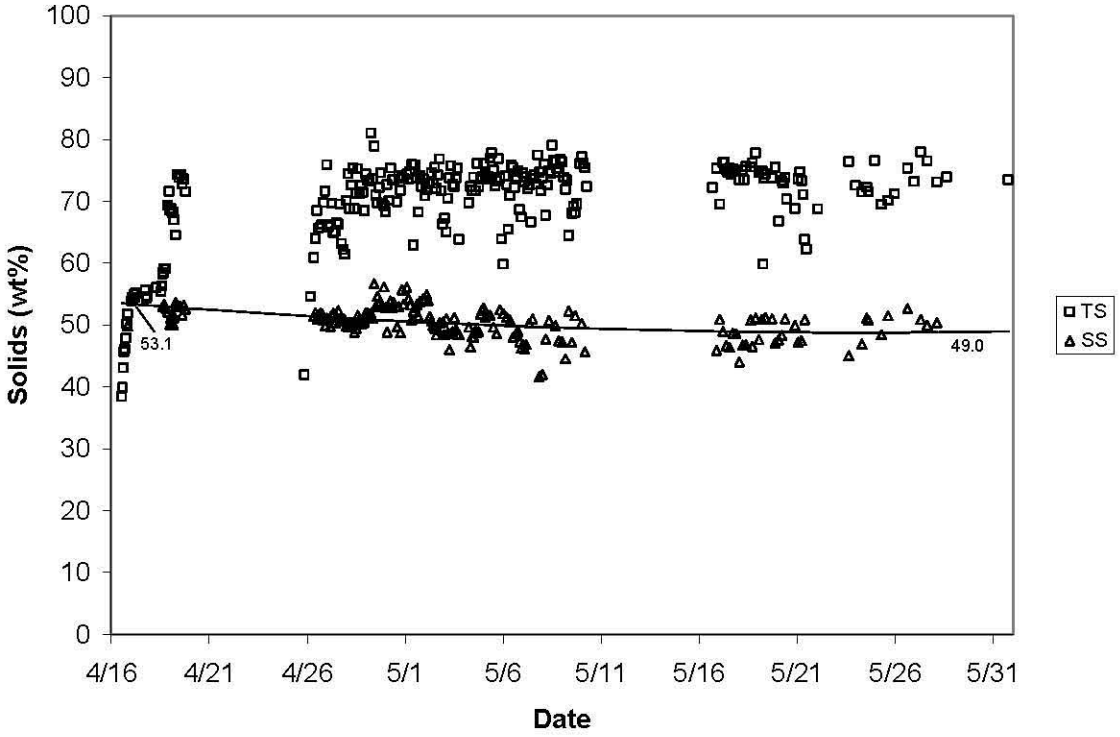
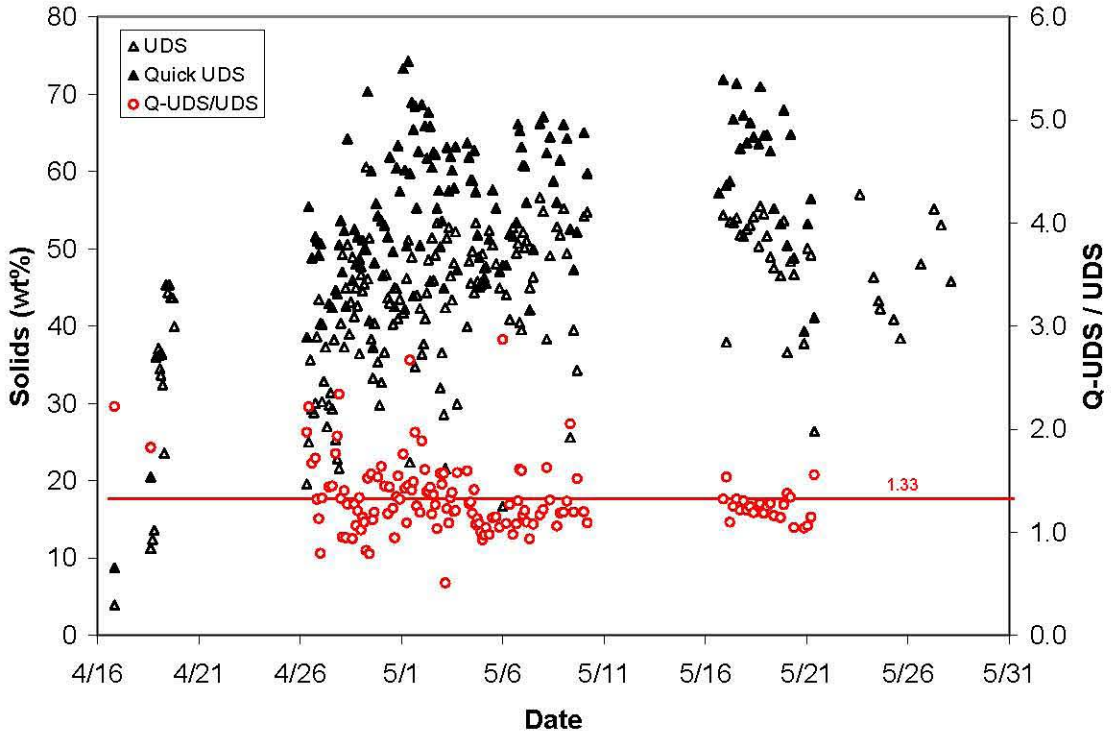


Figure 29. Slurry Recirculation UDS and Quick UDS Values



The only other sample type where more than a few TS measurements were made was the Product Dissolver (PD) samples. These data are shown in Table 11. The TS values varied from about 35 to 53 wt%.

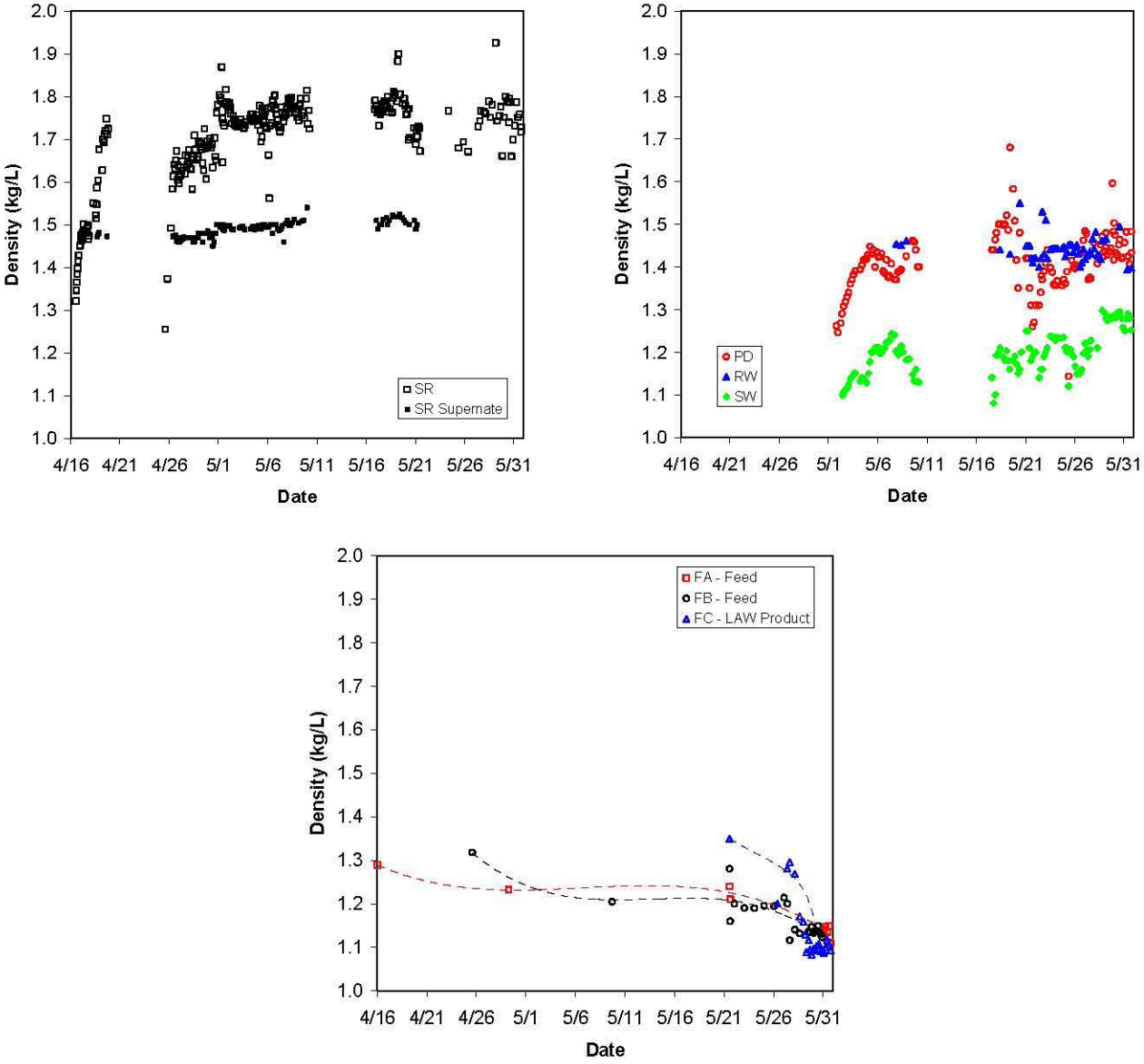
Table 11. Solids in Product Dissolver Samples

Sample	Total Solids (wt%)	Supernate Solids (wt%)	Undissolved Solids (wt%)
PD-0502-0900	35.28		
PD-0502-1300	36.83		
PD-0502-1700	37.90		
PD-0502-2100	38.96		
PD-0503-0100	39.96		
PD-0503-0500	42.23		
PD-0503-0900	46.21		
PD-0503-1300	46.21		
PD-0503-1700	45.57		
PD-0504-0500	44.48		
PD-0504-0900	43.63		
PD-0504-1300	48.13		
PD-0504-1700	48.13		
PD-0504-2100	48.81		
PD-0505-0100	50.21		
PD-0505-0500	52.50		
PD-0506-2100	48.54		
PD-0507-0100	44.57		
PD-0507-0500	43.69		
PD-0520-0600	42.25		
PD-0520-1000	44.15		
PD-0520-1400	44.84		
PD-0528-1700	55.06	44.82	18.56
PD-0531-1800	48.49		

The densities measured on process samples are shown in Figure 30. The SR samples generally were 1.7-1.8 kg/L, the SR supernate was about 1.5, the PD and RW samples were both around 1.4-1.5, and the SW samples were about 1.15-1.25, except for 5/28-5/31/08 where they were consistently 1.28 kg/L.

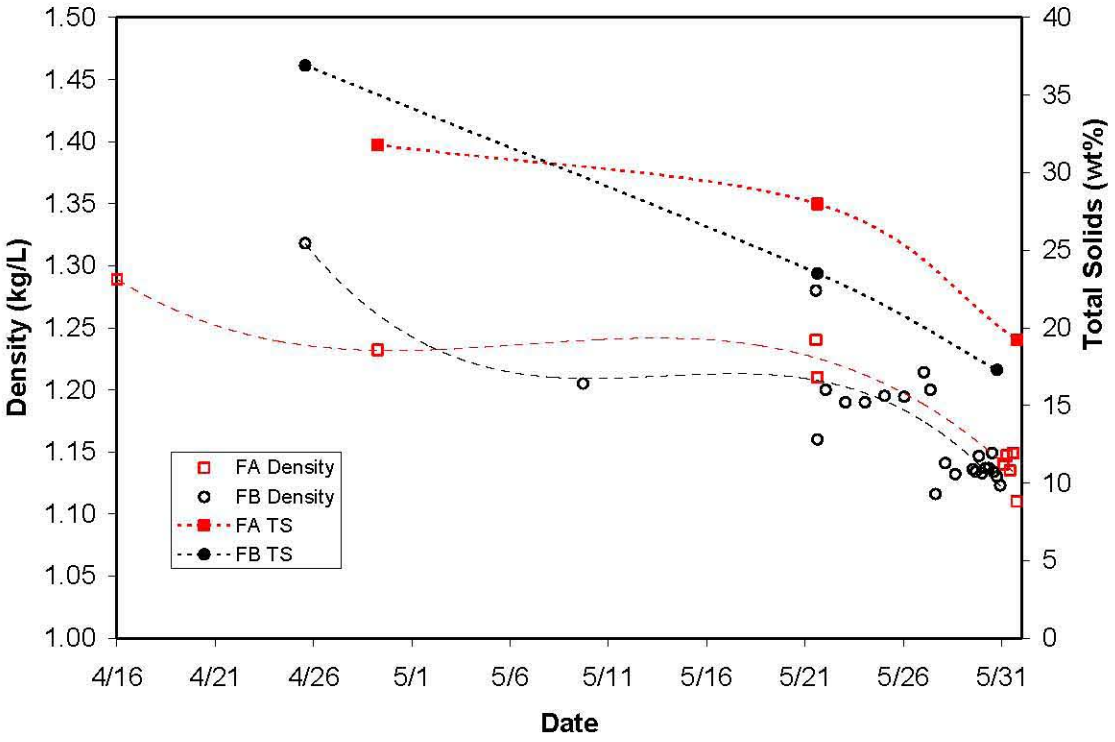
The feed samples (FA, FB) and LAW Product (FC) are also shown. The initial feed was about 1.30 kg/L; an initial tote sample was 1.289 and the initial FB sample was 1.318 kg/L. Figure 30 shows that these feed densities dropped as the tests progressed because the reconstitution scheme involved adding water to the products; more water was added than needed to exactly reconstitute, so the feeds became continually more dilute. Similarly, the LAW product in Tank C became more dilute, going from an initial ~1.3 to a final ~1.1 kg/L. Figure 30 shows both the density and total solids of Tanks A and B versus time.

Figure 30. Density of Process Samples



The density and TS of the feed samples FA and FB are both shown in Figure 31. The density and TS of each sample show similar downward trends.

Figure 31 Density and Total Solids of Feed Samples



On 5/27/08 at about 1800 hrs, the "steady-state" test was begun. Complete analyses were done on samples from Feed Tanks B and C (C was the LAW receipt tank), SR, CK, SW, RW, and the HLW product in Tank E (PR). From then until the completion of the testing, the CK samples were examined by PLM as needed and analyzed for elements and Cs at 4 hour intervals. The SW, HLW, and PD tanks and SR had density measured every 4 hours. One elements and Cs analysis was done on a PD sample each day. An SR sample was analyzed for elements and Cs every 4 hours.

Figures 32-37 show plots of the CK, SR, PD, SW, feed FB, and product FC LAW and Tank E HLW elemental analyses. These data are shown from 5/21/08 to 6/1/08, except for the feed and products which are shown from 4/25/08 to 6/1/08.

The data in Figure 32 show that the Na, K, and Cs in the cake samples stayed relatively constant. There are a few higher Cs values after 5/29/08, but these may be incorrect analyses; the Cs results for the last several days of operation were much more inconsistent than for the previous days. The Al and P both appear to have decreased.

The limited number of SR samples taken indicates that the composition did not change significantly, as shown in Figure 33. The small amount of data and the scatter make it difficult to determine if there were any trends in the compositions.

Figure 34 indicates that the product dissolver Al and K concentrations both decreased starting on 5/21/08, which is consistent with the implementation of the full washing cycle in the centrifuge. The Cs concentration increased after introduction to

the feed until it reached a steady value of about 8 mg/L. The Na concentration remained steady as expected. Note the final data points on 6/1/08 are probably incorrect.

The few SW samples shown in Figure 35 indicate that the concentrations of all species may have gone up slightly. The sum of the washes data shown for 5/30/08 agrees with what would be expected. All species in the sum of washes 1-4 are at higher concentrations than in washes 5-8.

The RW samples (Figure 36) show that all species except Cs remained at approximately constant values. The Cs increased as expected after it was added to the feed and appears to have reached a steady concentration of about 250-300 mg/L.

Figures 37 and 38 show that all of the feed species in Tank B decreased over time due to the dilution that occurred during reconstitution. For the time period from 5/27/08 to 5/31/08, the concentrations of all Tank E HLW species increased with time, except P which remained approximately constant. For the Tank C LAW product, all species concentrations decreased over this same time interval.

Figure 32. Elemental Analyses of Centrifuge Cake Samples

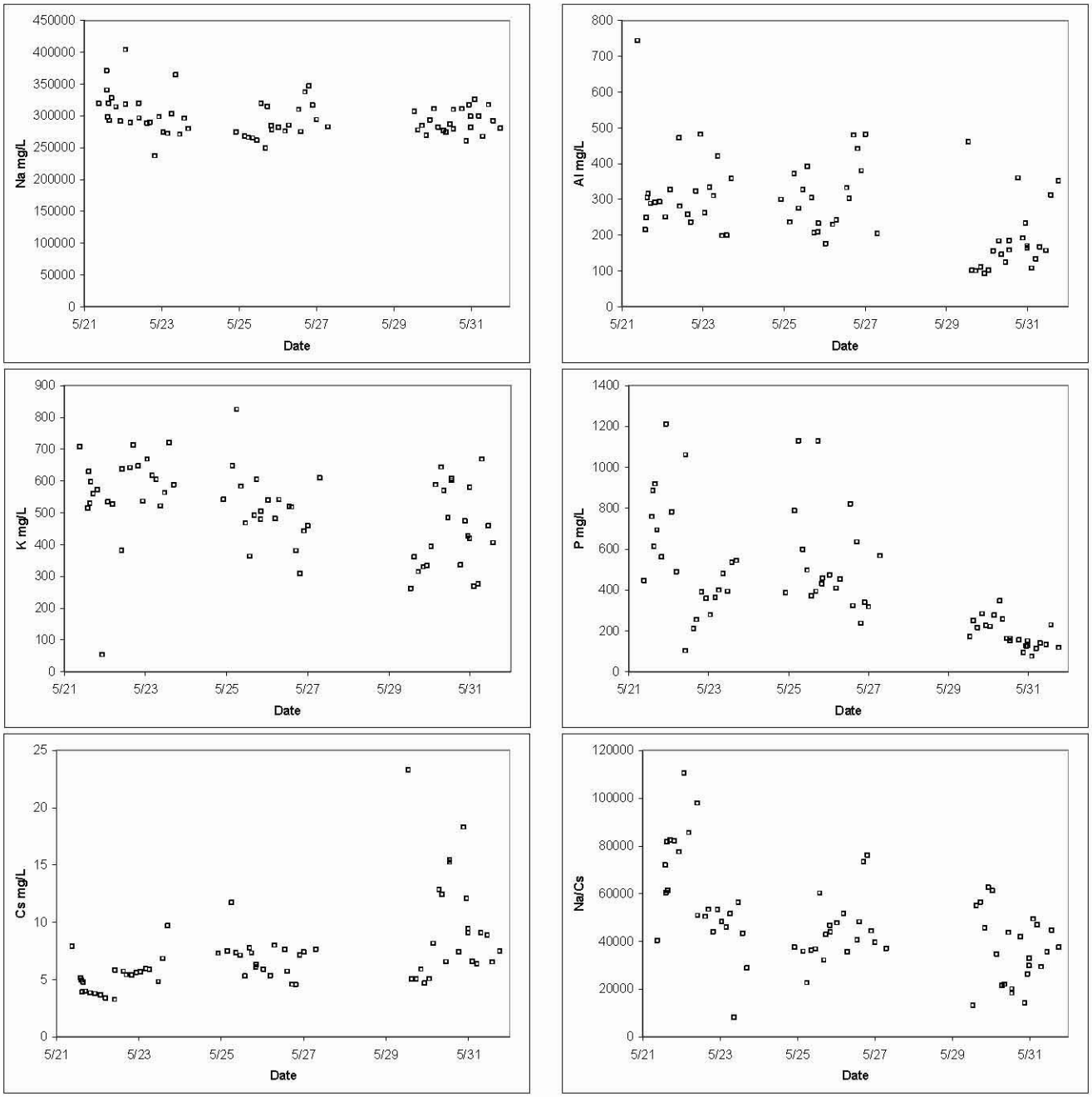


Figure 33. Elemental Analyses of Slurry Recirculation Samples

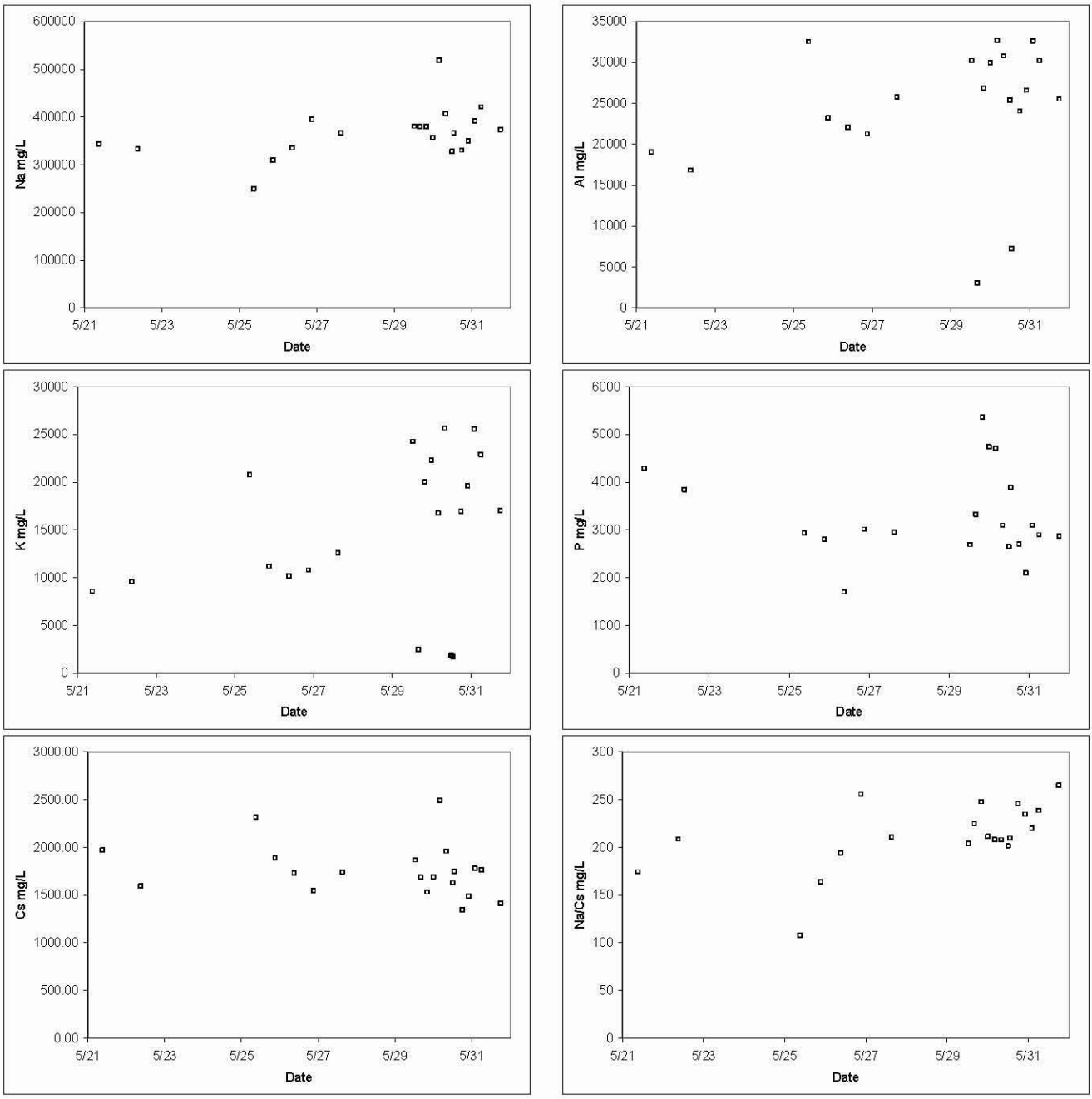
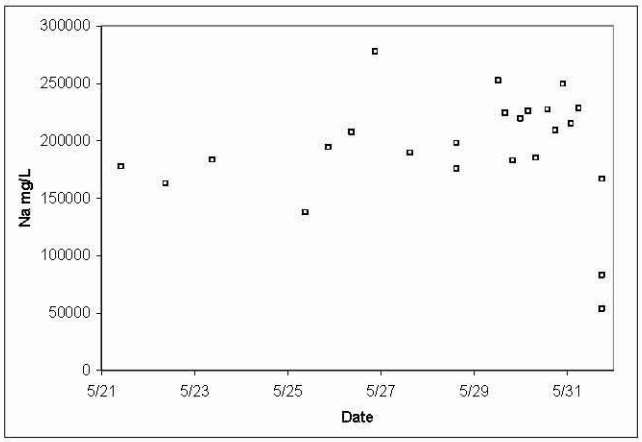
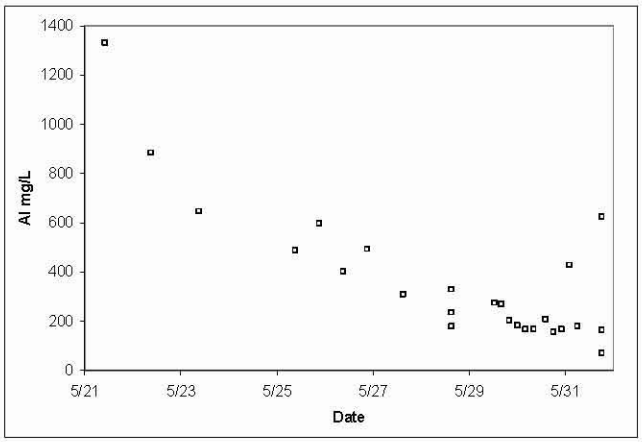
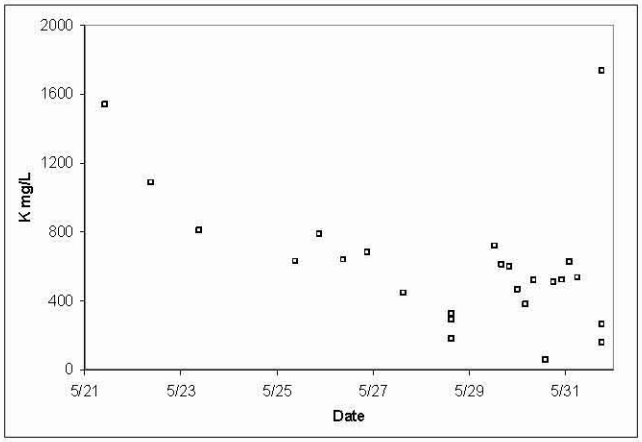
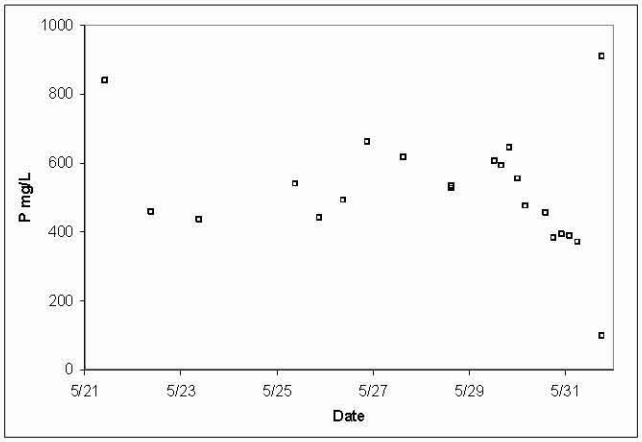


Figure 34. Elemental Analyses of Product Dissolver Samples









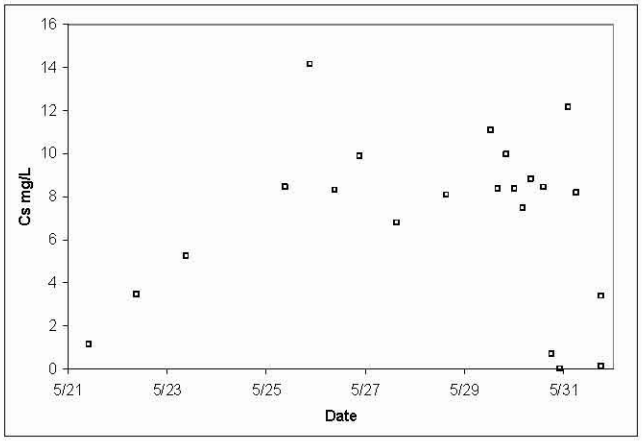
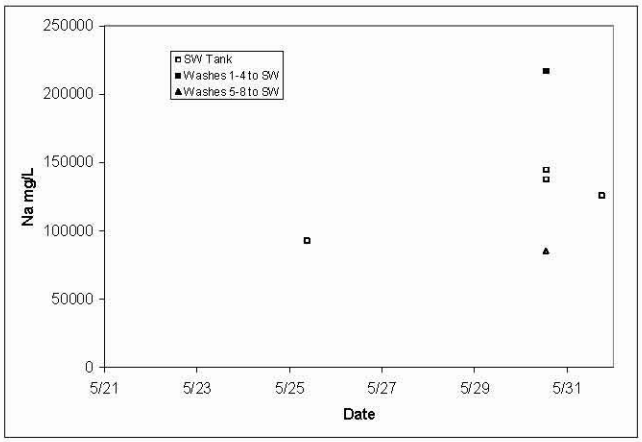
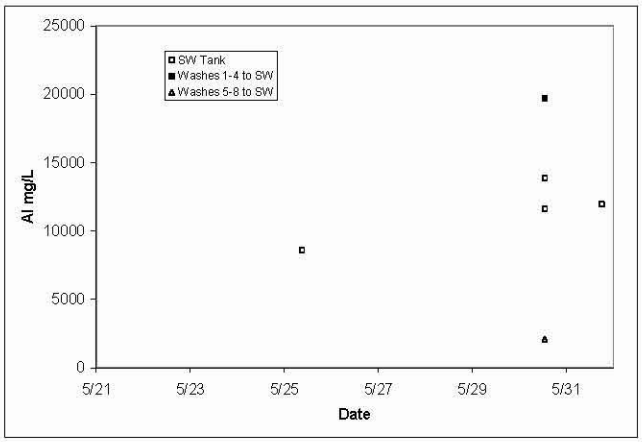
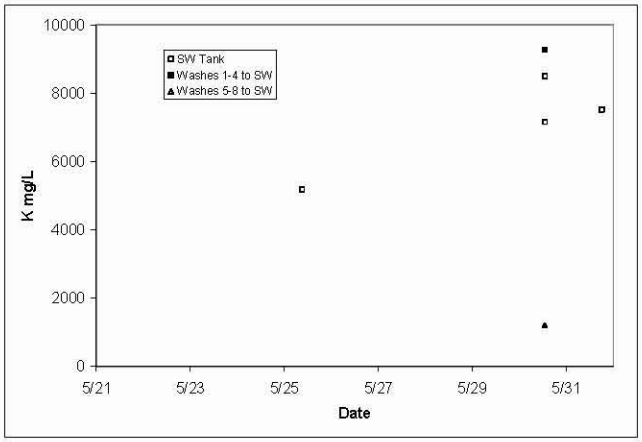
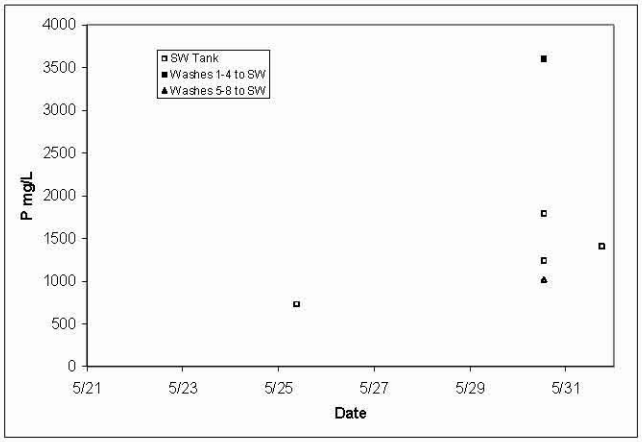


Figure 35. Elemental Analyses of Spent Wash Samples









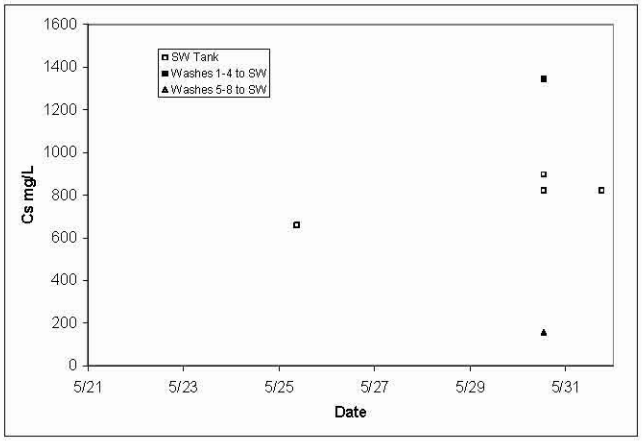
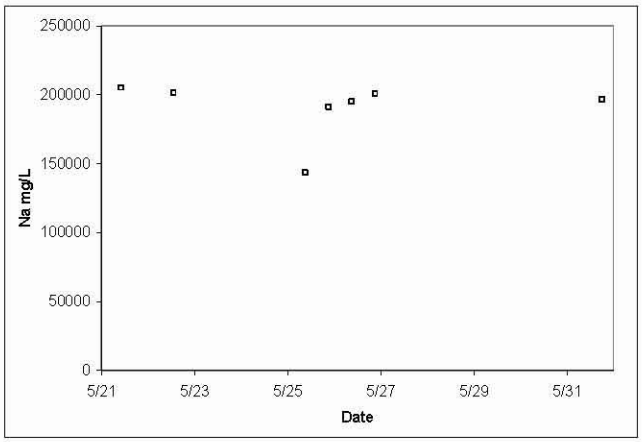
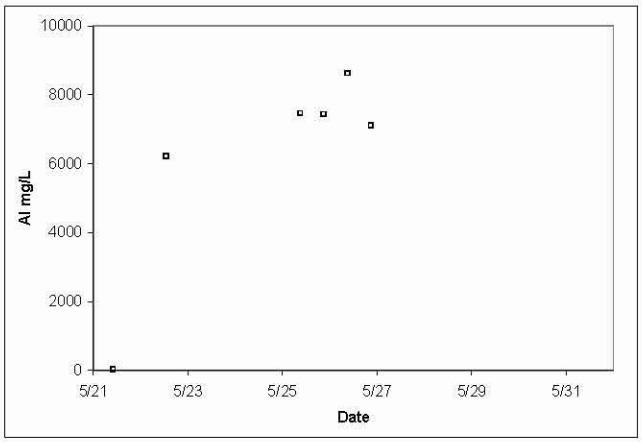
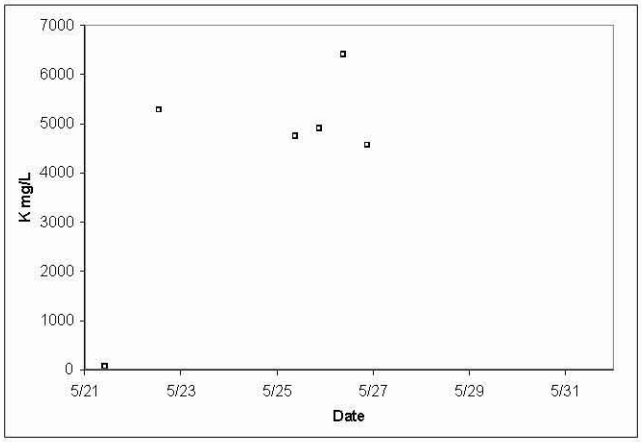
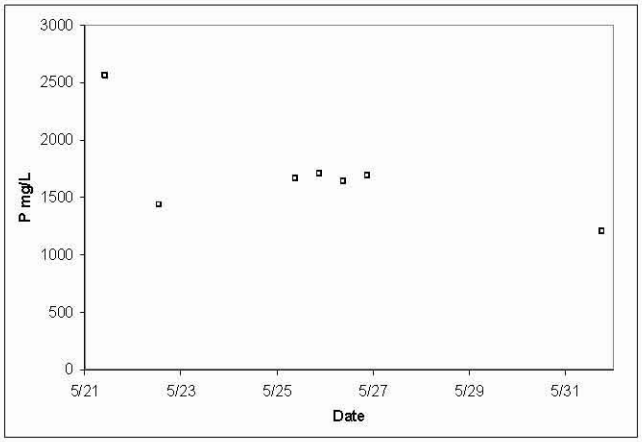


Figure 36. Elemental Analyses of Recycle Wash Samples









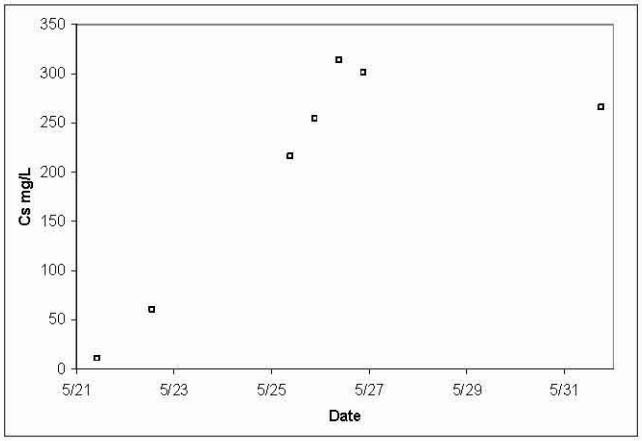
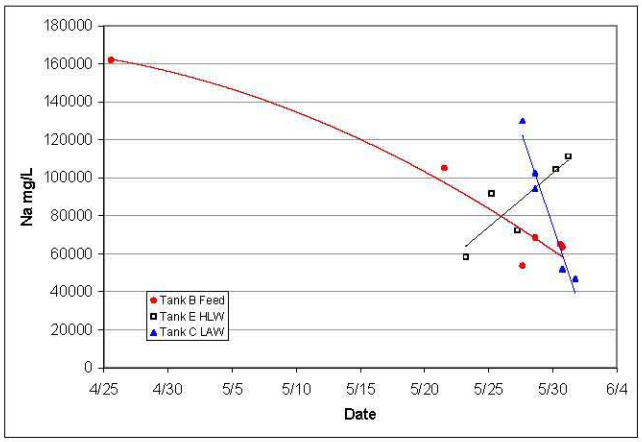
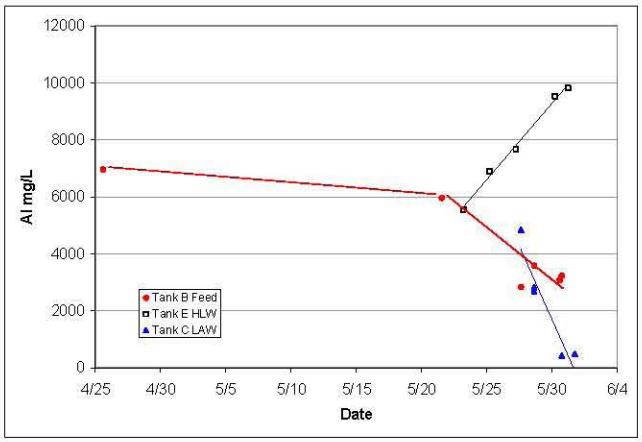
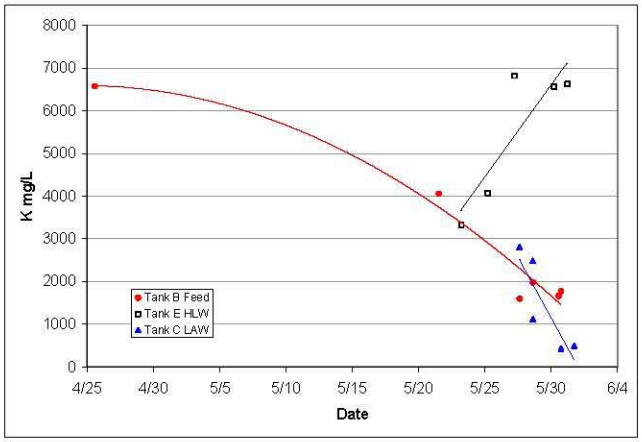
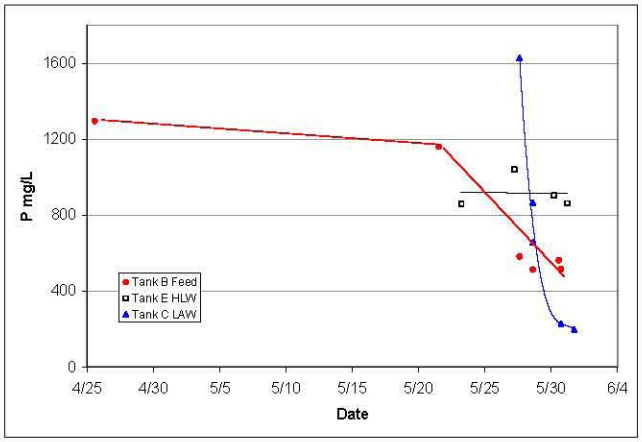


Figure 37. Elemental Analyses of Feed and Product Samples









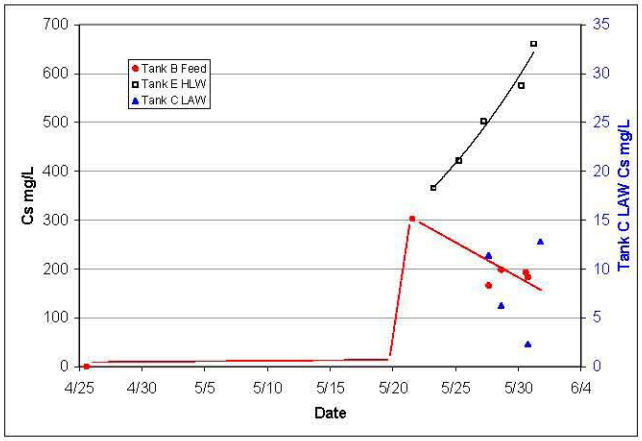
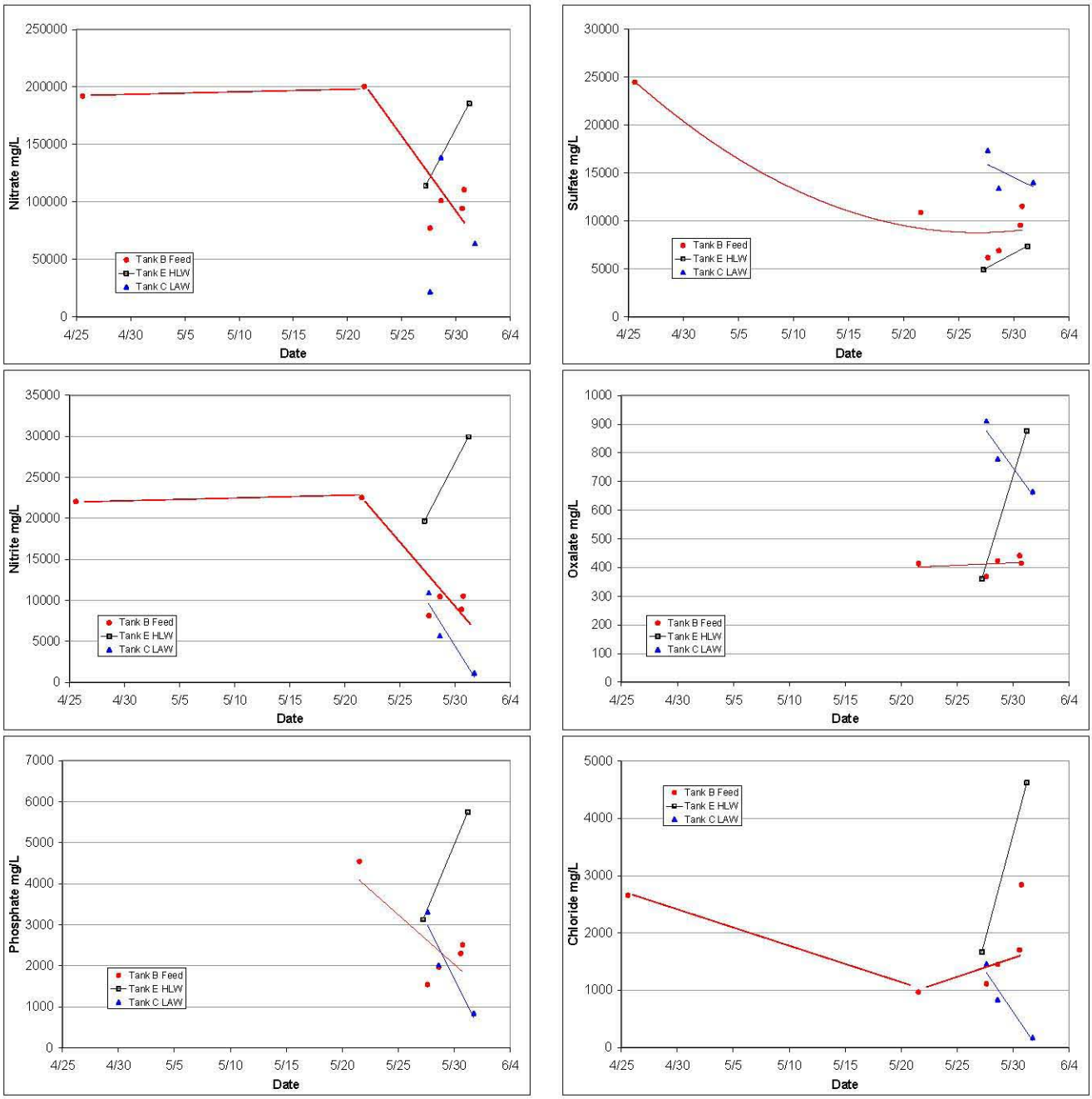


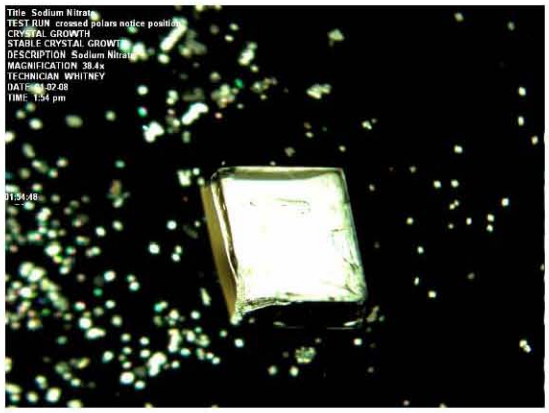
Figure 38. Anion Chromatography Analyses of Feed and Product Samples



4.2.5.1 PLM Results

PLM was used throughout the SST test campaign to analyze salt crystals for the effects of changes in Crystallizer operating conditions. Standards of SST simulant with various salt crystals were prepared by Whitney Thomas and a hard copy catalog developed of slide pictures for reference in file labeled FC PLM Crystal Examples provided with training for FC operations. The crystals were grown in pure binary solutions (water + salt). An example of reference crystal for sodium nitrate is sodium in Figure 39.

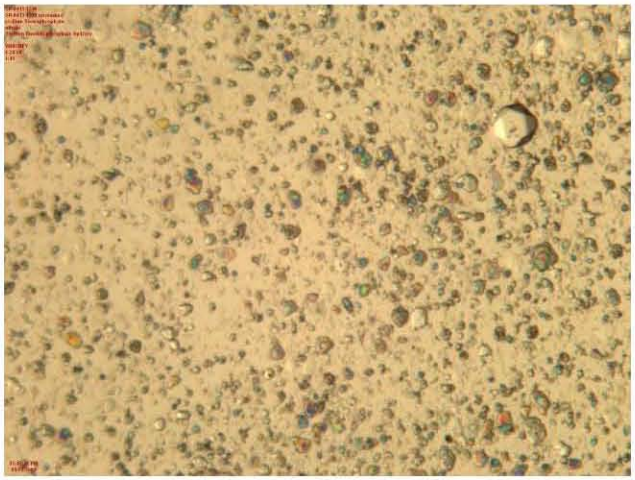
Figure 39. Reference PLM of Sodium Nitrate Crystal.



The reference sodium nitrate crystal shows sharp edges indicating steady growth conditions.

Feed Tank Samples were analyzed for the presence of undissolved salt crystals in the simulant as shown in Figure 40.

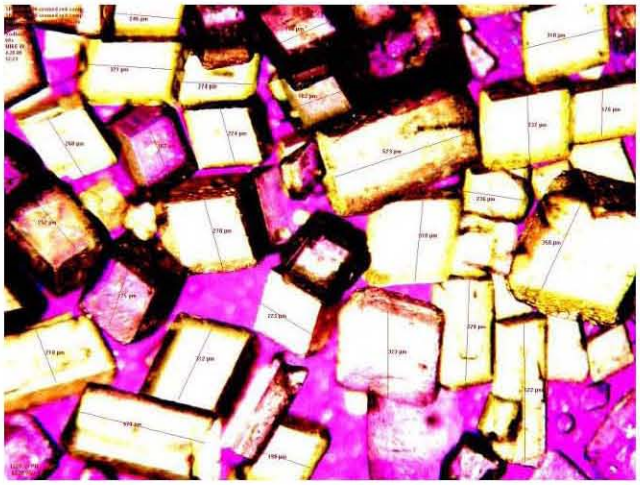
Figure 40. Undissolved Salts in Feed Sample.



During storage at cold conditions (<50ºF), sodium nitrate and sodium fluoride-phosphate crystals formed in the feed simulant. Sodium nitrate dissolved upon warming to room temperature (70ºF), while some sodium fluoride-phosphate remained as a metastable solid in the feed tanks.

During crystallizer operation, the slurry samples analyzed showed sodium nitrate crystals growing to the size of several hundred microns. A good example of the large sodium nitrate crystals can be seen in Figure 41.

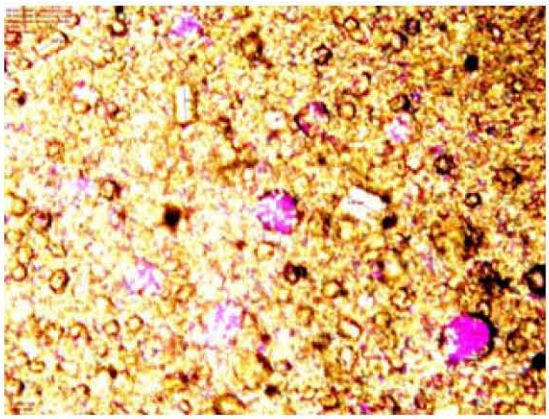
Figure 41. Large Sodium Nitrate Crystals in Crystallizer Slurry



In this sample, large sodium nitrate crystals had edge lengths of 200-500µm. This indicates a large mean crystal size and narrow size distribution that allows efficient solid/liquid in the centrifuge.

When the crystallizer was operated at suboptimal conditions, due to pressure and temperature fluctuations and/or water dilution, the edges of the sodium nitrate crystals were rounded, and the average crystal size was reduced as shown in Figure 42.

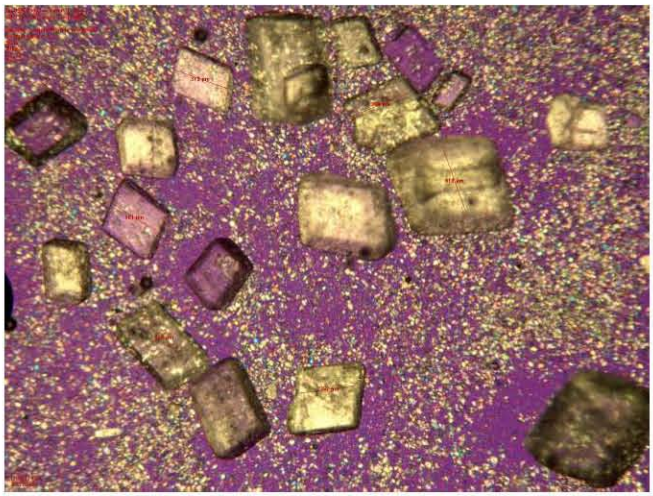
Figure 42. Crystal Matrix with Poor Morphology



In this sample, crystal crystalline edges are poorly defined, small mean crystal size (<100 µm) and wide size distribution. This slurry "mush" exhibits poor solid/liquid separation in the centrifuge.

Most frequently, the crystals exhibited a bimodal size distribution as shown in Figure 43.

Figure 43. Typical Bimodal Crystal Size Distribution

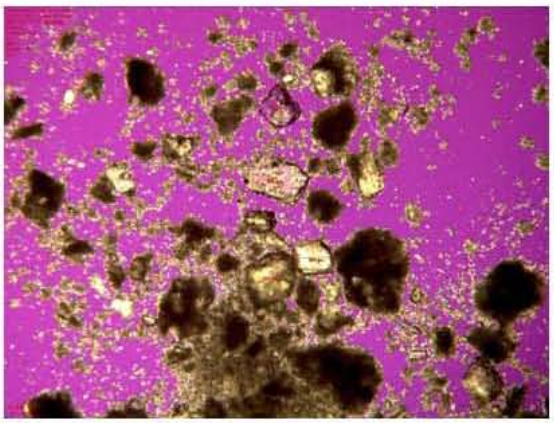


The bimodal size distribution is partly caused by the differing crystal growth rates of large sodium nitrate (fast) and small sodium carbonate and sulfate (slow). However, this sample exhibits rounded crystal edges and excessive nucleation of fine crystals.

These forms are indicative of temperature cycling; fine crystals form upon cooling and edges dissolve upon heating or dilution. This particle size distribution separates poorly in the centrifuge. Large crystals do not capture small particles in the cake and small crystals flow through the centrifuge screen or form a low-permeability rind around the outside perimeter of the cake. This stratification requires frequent backwashing of the centrifuge to remove the rind and restore proper operation to the centrifuge.

Samples of centrifuge cake typically exhibited smaller crystal size distribution than the crystallizer slurry samples as shown in Figure 44.

Figure 44. Typical Centrifuge Cake Sample



Compared to crystallizer slurry, the centrifuge cake typically contained a much higher fraction of fine crystals. Fine crystals in the cake may be caused by *in situ* dissolution of large crystals by unsaturated wash liquor or recycle of fines from unfiltered wash solution. Due to the high fraction of fines in this sample, this type of centrifuge cake exhibited poor liquid/solid separation and required frequent centrifuge backwashing to remove the low permeability rind.

Because small crystals agglomerate to form larger particles, screen analysis of crystal size distribution is often inaccurate in determining actual crystal size distribution of slurry and centrifuge samples. PLM analysis allowed visual analysis of crystal type, size distribution, and morphology. Thus, PLM analysis was helpful in determining the causes of poor solid/liquid separation in the centrifuge.

4.2.6 Sodium Aluminosilicate (NAS) Gel in Simulant

During FC Pilot Plant operation, a brown gel was observed in a retain sample of the simulant. Centrifuge cake, product, and purge samples also contained brown gel. Upon standing, the brown gel gradually settled out of feed material that was reconstituted from product and purge streams. This material was not observed in laboratory simulant or actual waste experiments. The settled brown gel layer in Tank A is shown in Figure 45.

Figure 45. Brown Gel Settling in Tank A

The gel layer was sampled from the tank and filtered for analysis as shown in Figure 46.

Figure 46. Brown Gel Filtration

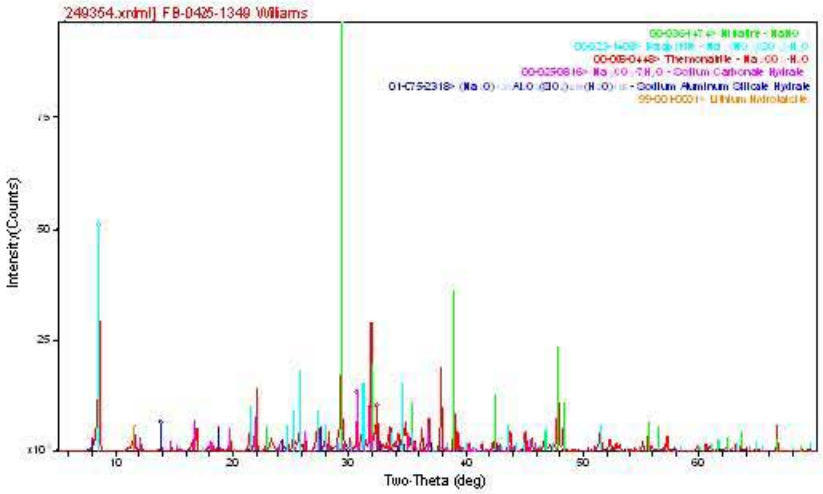
However, the gel filtered poorly, and after four days of vacuum filtration, less than one-half of the sample passed through the filter, thus the gel was unsuitable for XRD analysis. Another sample of the gel was leached in sodium hydroxide to determine if the gel was precipitated alumina. The caustic leached sample is shown in Figure 47.

Figure 47. Caustic Leached Gel

As shown above, the caustic leach of the gel did not improve the filterability of the material or allow XRD analysis.

Another portion of the gel sample was heated, followed by a lithium nitrate addition. The gel solution decomposed to a thin slurry that filtered rapidly. XRD analysis of the filter cake is shown in Figure 48.

Figure 48. XRD of Lithium Hydrotalcite/Hydroxy Sodalite



XRD analysis of the filter cake identified the presence of sodium salts ($NaNO_3$, $Na_3NO_3SO_4.H_2O$, $Na_2CO_3.1H_2O$, $Na_2CO_3.7H_2O$) from the waste simulant liquor, the formation of Lithium Hydrotalcite $Li_2CO_3.4Al(OH)_3.3H_2O$ precipitated from lithium nitrate and alumina, and Sodium Aluminum Silicate Hydrate (likely hydroxy sodalite - $Na_8Al_6Si_6O_{24}(OH)_2•2H_2O$).

ICP analysis of the feed retained sample indicated 17ppm silica in the supernatant. The origin of the Si is unknown but was suspected to originate as impurities in the original simulant chemicals. ICP analysis of the brown layer indicated roughly equal amounts of sodium, aluminum, and silicon.

Based on the ICP and XRD analysis, the gel was initially comprised of a very small amount (<<1%) sodium aluminosilicate (NAS) gel ($Na_5Al_6Si_7O_{25½}•15½H_2O$) that contained large inclusions of simulant liquor. During settling, the gel coalesced by reducing liquor inclusions.

As demonstrated by the slow filtration of the sample, NAS gel in very low concentrations can be very problematic to waste treatment. NAS gelation can result in filter and line pluggage as well as poor product quality due to inadequate solid/liquid separation.

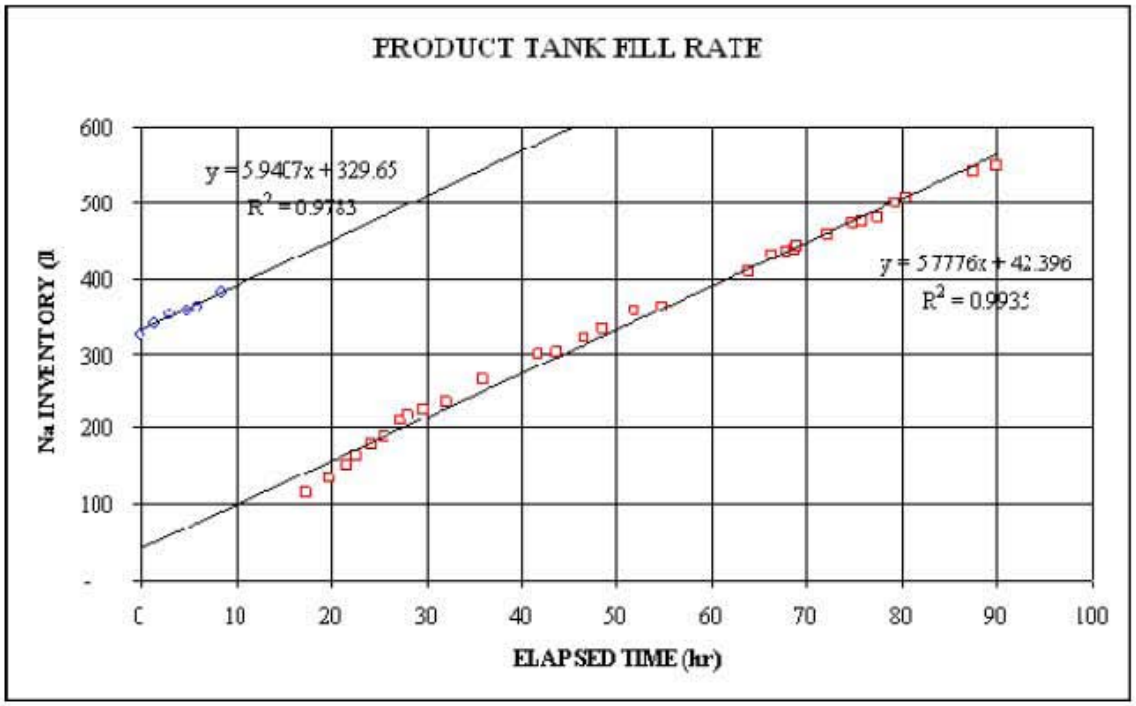
To reduce NAS gel in the crystallizer system, feed tanks containing brown gel layers were isolated. Clear supernatant was decanted from the tops of the tanks for recycle to the process, and brown gel layers were exported to tote bins.

4.2.7 Sodium Yield Calculations and Comparison to Flowsheet Yield

Based on the tank inventory and specific gravity data, estimated Na yield for the period 5/28 - 5/31 was approximately 52%. The estimate is based upon the relative rates-of-gain of sodium inventory in the Product Tank C and Purge Tank E.

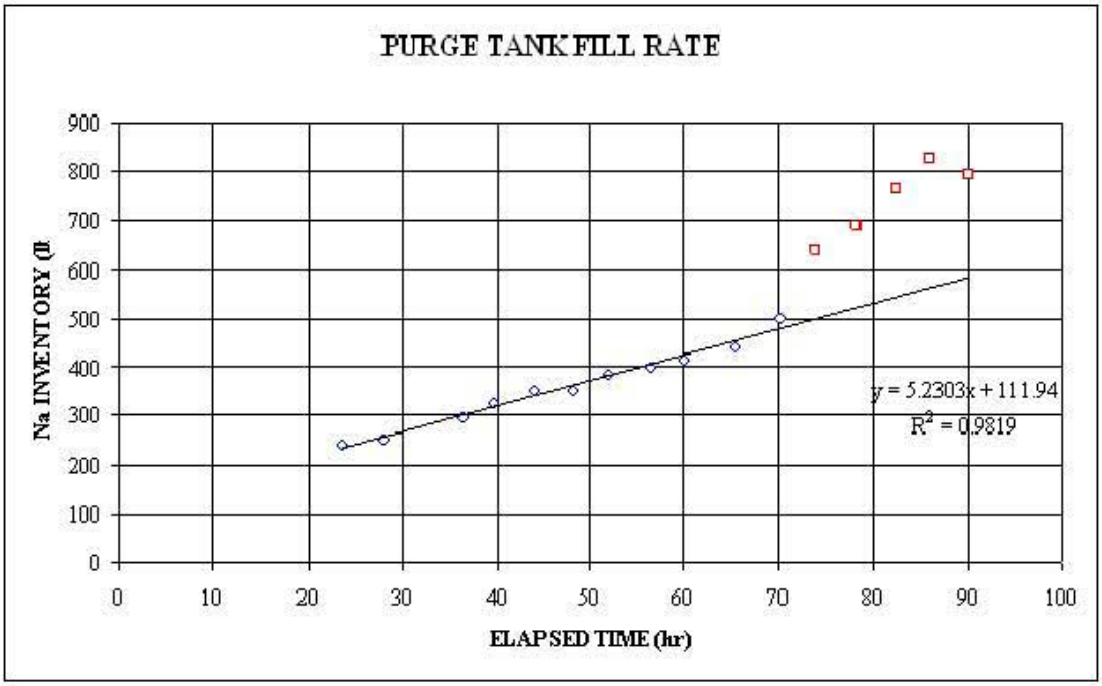
The Na rate-of-gain in Product Tank C is shown by the slope of the lines in Figure 49 and averaged 5.8 lb Na/hr over the period.

Figure 49. Product Tank Fill Rate



The Na rate-of-gain in Purge Tank E is shown by the slope of the line in Figure 50 and averaged 5.2 lb Na/hr. The final 5 data points were discontinuous and were omitted from the rate calculation.

Figure 50. Purge Tank Fill Rate



Based on the relative rates, the Na yield is Product/(Product + Purge) = 5.8/(5.8+5.3) = 52%

The sodium feed rate from Feed Tanks A & B averaged 9.7 lb Na/hr as shown in Figure 51.

The Na Product + Purge rate (5.8 + 5.3 = 11.1) exceeds the Feed Rate (9.3 lb/min) because the sodium inventory (as indicated by specific gravity) in the crystallizer was decreasing at an average rate of -1.96 lb Na/hr as shown in Figure 52.

Figure 51. Sodium Feed Rate

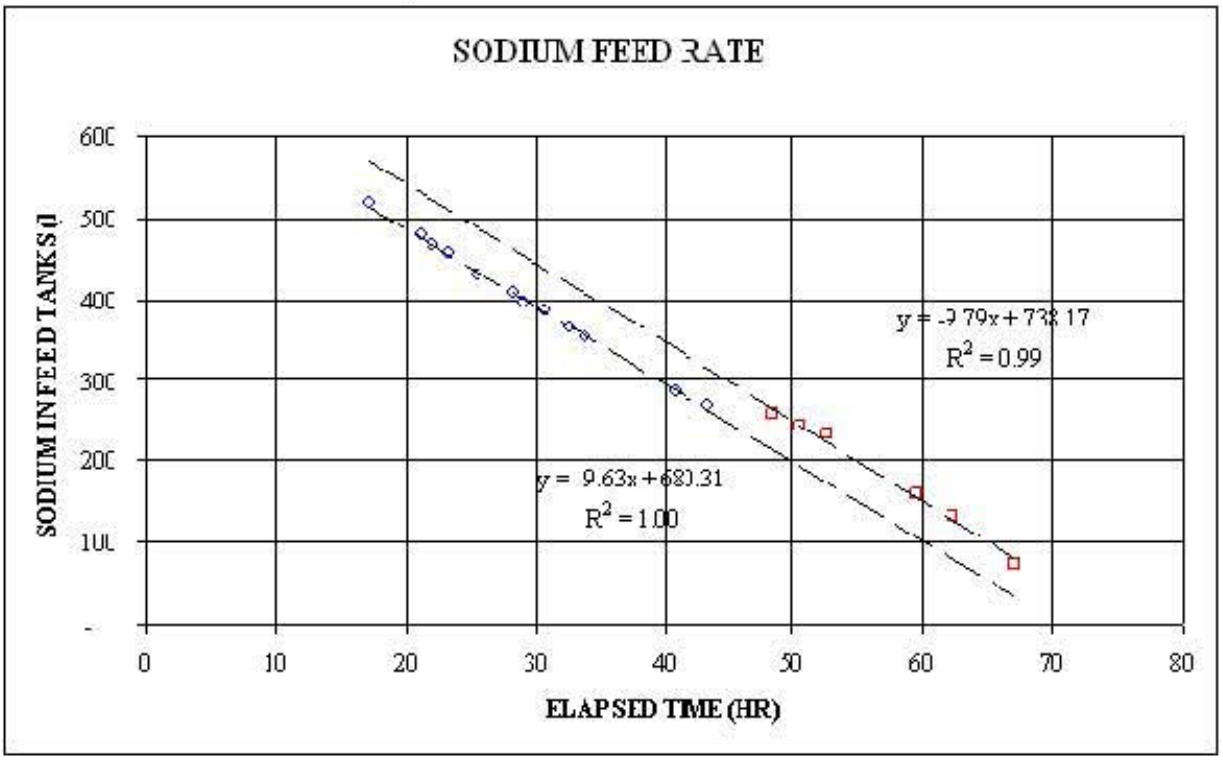
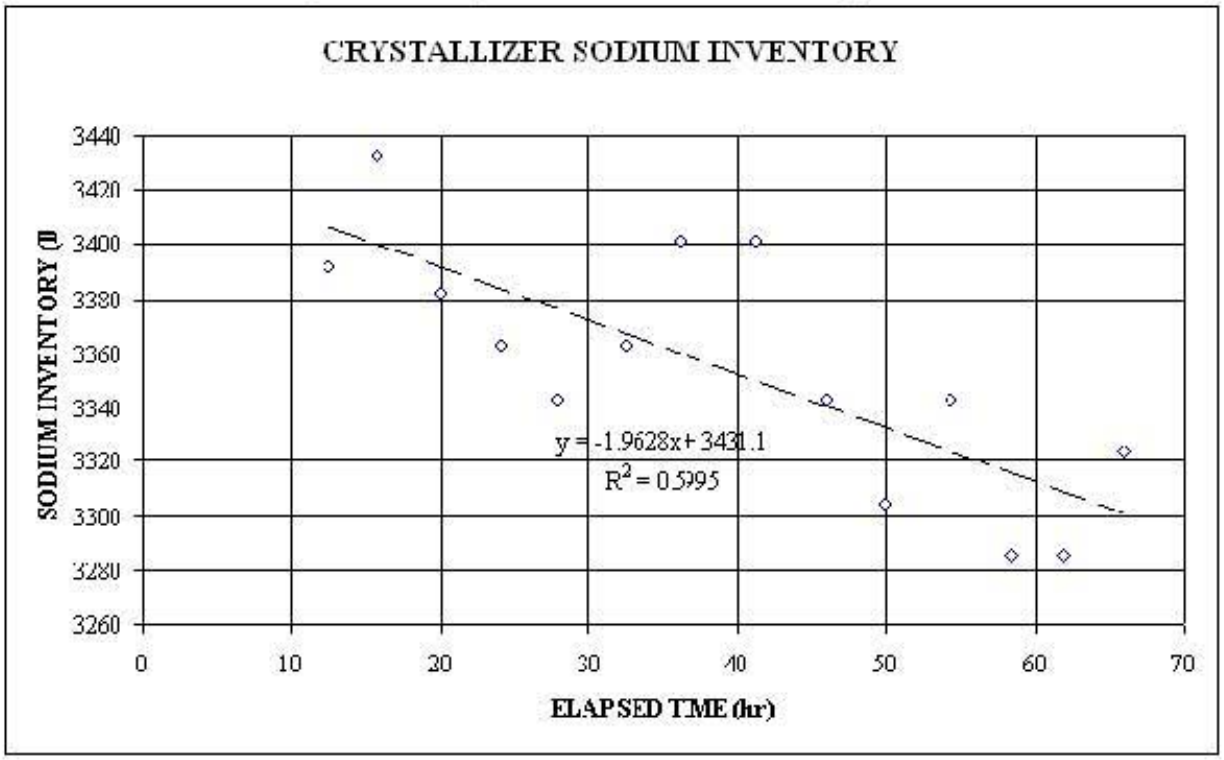


Figure 52. Crystallizer Sodium Inventory

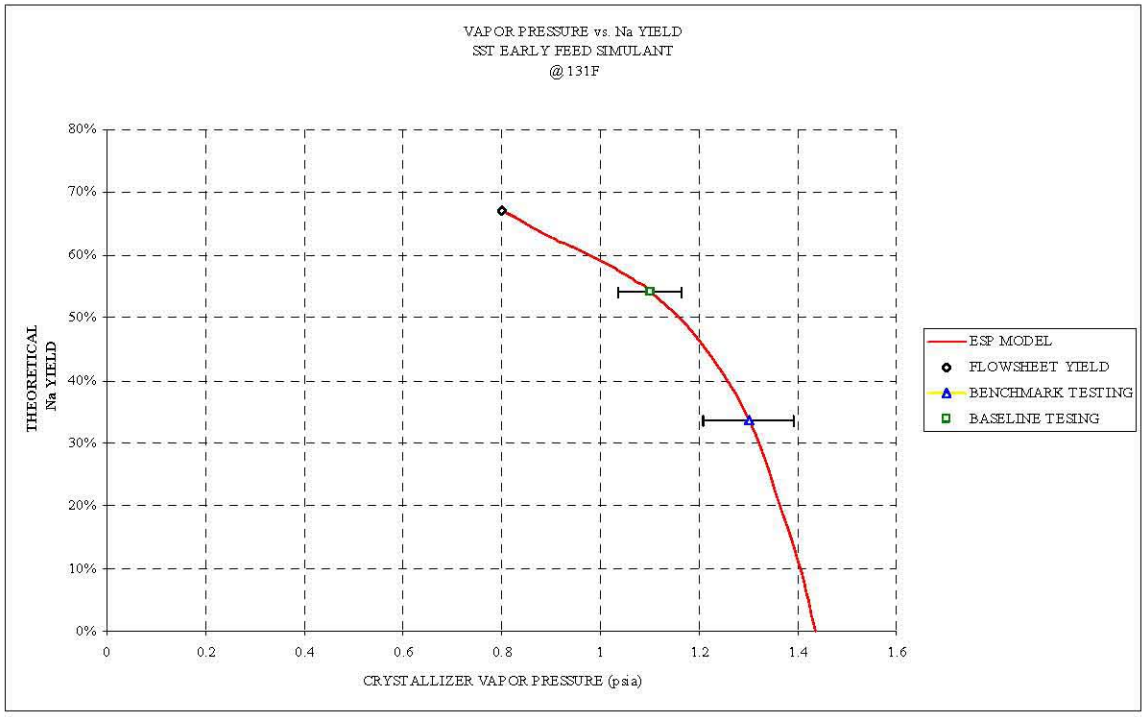


Total In = 9.63 + 1.96 = 11.6 Na/hr
Total Out = 5.8 + 5.3 = 11.1 Na/hr

Difference (11.6 – 11.1)/11.6 = 4%

For a specific feed composition (e.g. SST Early) a correlation between the sodium yield and crystallizer vapor pressure at constant temperature is possible, since the vapor pressure decreases with the extent of evaporation and the sodium yield is proportional to the solids fraction (solids/total slurry). This correlation assumes that all solids formed in the crystallizer are separated in the centrifuge. A graph of the correlation of crystallizer vapor pressure at 131°F to sodium yield is shown in Figure 53.

Figure 53. Correlation of Crystallizer Vapor Pressure to Sodium Yield at 131ºF



During benchmark testing, liquor was not recycled from the spent liquor tank to the crystallizer. By this method, the crystallizer acts as a one-pass crystallizer where the yield is limited by the maximum slurry density. During benchmark testing, crystallizer pressure averaged 1.3 psia at 131ºF, which corresponds to a 34% sodium yield.

During baseline testing, liquor was recycled from the spent wash tank to the centrifuge. This lowers slurry density in the crystallizer and allows a greater extent of evaporation. During baseline testing, crystallizer pressure averaged 1.1 psia at 131ºF, which corresponds to a 54% sodium yield. This value agrees with the mass balance yield calculated above (52 $\pm$ 4%).

Flowsheet sodium yield was 67%. This yield was demonstrated in two-stage laboratory tests, and it is possible in a continuous system at higher liquor recycle rates and a greater extent of evaporation. At the flowsheet sodium yield, crystallizer vapor pressure is 0.8 psia at 131ºF.

5.0 LESSONS LEARNED

One of the most important aspects of any pilot-scale testing is to identify lessons learned during pilot scale design, construction, and operations because they may be used for scale-up. In nuclear facilities, this is even more important since design changes and maintenance issues become very challenging in a full-scale facility.

5.1 Hardware

5.1.1 Centrifuge

The single most important piece of hardware that needs upgrade is the centrifuge. Many process upset conditions resulted from the mismatch of centrifuge capacity to the rest of the system as elaborated below.

5.1.1.1 Centrifuge Capacity

The centrifuge capacity turned out to be much lower than the production rate of the crystallizer. Cycle time for centrifuge operation was approximately 16 minutes. The process flowsheet was based upon the assumption that the cycle time would be 4 to 5 minutes. Longer cycle times resulted in lowered production capacity.

Centrifuge throughput was low due to high fines loading because of temperature cycling of the crystallizer and the recycle of fines from the product dissolver due to failure of the cross-flow filter. Fine particles, high rotational speed, and long cycle times caused centrifuge cake to compact, thus lowering cake porosity and the extent of deliquoring and decontamination. As a result, the centrifuge required frequent heel removals to clean the screen and improve cake permeability for successive cycles.

Additionally, the centrifuge was operated conservatively (thinner cake thickness) to minimize or limit feed overflow into the product tank. More-frequent-than-expected heel removals also reduced capacity. Thus, the combined effect was that the centrifuge capacity was about 5 – 8 times lower than the crystallizer and balance of the pilot facility.

5.1.1.2 Product Chute Pluggage

The centrifuge peeled product discharged by gravity through a 4 inch diameter product chute which penetrated through the centrifuge door. The peeler knife and other internal hardware leading to the product chute were configured in a way that peeled wet cake tended to buildup at the mouth of the chute. After about a dozen centrifuge cycles, the chute entrance plugged and required extensive washing and clearing. If the crystal cake was not deliquored completely, the pluggage occurred earlier.

Chute pluggage was the primary reason that the spin cycles were long and the overall cycle time was much longer than anticipated. Long cycle time resulted in cake compaction and lower cake permeability. Thus, by increasing the cycle time and extent of deliquoring, the frequency of backwashing the screen also increased, thereby lowering the production rate.

Larger (production-scale) centrifuges are equipped with a screw auger that is essential to preclude the pluggage experienced in the pilot scale tests.

5.1.1.3 Centrifuge Cleaning

Due to the pluggage described above, a considerable time was spent on cleaning the centrifuge through the product chute after pluggage. This also caused process-upset conditions because a lot of condensate was added for cleaning. The excess condensate diluted the in-process tanks, as well as the product and reconstituted feed. A diluted feed dissolved slurry in the crystallizer and potentially reduced crystal quality.

Large pharmaceutical centrifuges have self-cleaning hardware, thus minimizing the amount of wash liquids. Additionally, the centrifuge should have PLC control of speed for heel removal.

5.1.2 Crystallizer

5.1.2.1 Crystallizer Control

The crystallizer's function was to concentrate the slurry to the desired density while maintaining the temperature within a narrow operational range. The temperature was controlled by the system vacuum. The crystallizer vacuum was controlled by bleed air into the vacuum system.

The bleed-air control valve was undersized for the application. To increase the bleed air rate, compressed air was connected to the bleed valve and the compressed air was manually controlled by a mass flow controller. However, the air controller required continuous monitoring and adjustments. Stable pressure control was not attained, and crystallizer pressure and boiling temperature fluctuated erratically.

Because of temperature fluctuations, crystal size distribution in the crystallizer varied from sample-to-sample. When the temperature dropped, fine crystals nucleated; when the temperature rose, coarse crystals dissolved. As a result of the irregular size distribution, the non-uniform slurry was difficult to process in the centrifuge.

It is recommended that the crystallizer pressure control system be upgraded before further testing.

5.1.2.2 Sampler Hardware

Initially, a ½" valve and a length of pipe were installed to pull slurry samples from the slurry loop. This arrangement resulted in frequent pluggage. Later on, another sampling scheme with condensate flush line was implemented with reasonable success. It is recommended that an Isolok type of sampler be installed prior to further work.

5.1.3 Pumps

5.1.3.1 Double Seal Pumps

The existing process tank pumps were selected based upon the information that there will be no solids in the process tanks. However, during the testing, it was observed that the process tank did have solids as testing progressed. This caused two process tank pump failures. Future testing will need pumps with double seals and clean flush liquids.

5.1.3.2 Double Diaphragm Pump

The existing double diaphragm pump in the Feed/Receipt Tanks area caused a great deal of vibration in the associated piping. This pump should be replaced by a large capacity centrifugal pump with priming capability.

5.1.4 Heaters

The electric heaters for the process tanks required more power to reduce the time to reach process temperature. As a result, the heater fuses frequently blew, and the wash solutions did not achieve the required process temperature. Additionally, the heater controls need to be reevaluated for a trouble free operation.

5.1.5 Instrumentation

Several instrumentation systems need re-evaluation or improvements as listed below.

- Replace level instrumentation for process tanks
- Use diaphragm type pressure gauges on process tanks
- Re-evaluate deaereators on crystallizer level and density measurements
- Lower PD density pressure tap
- More accurate steam flow measurement

5.1.6 System Configuration

Prior to further testing on the FCPP many system configuration changes will be needed as provided below.

- Need additional condensate flush lines
- Consider circulating loop with small draw-off for feed and SW recycle
- Consider automatic SW flush of centrifuge feed line
- Provide double valve isolation or other hardware changes to facilitate lock & tag
- Consider additional proportional flow control valves

5.1.7 Controls

For a complex system like this facility, it is very important for various control functions to operate properly. Otherwise, it becomes very labor intensive to monitor and adjust parameters manually. The following control functions need to be reevaluated.

- Better evaluation of control loops
  - Automatically adjust bleed air to crystallizer temperature
  - Automatic control of crystallizer level by feed rate
  - Automatic control of reboiler steam
- Working DAS PID controls
- Robust process tank heater controls

5.1.8 Reboiler Steam Supply

The original design of the desuperheater in the steam supply for the reboiler used a single conical spray nozzle to inject water into the 6" steam supply pipe downstream of the control valve that dropped the pressure from 125 psig to about 3 psia. It was observed that the conical spray nozzle did not work above about 5 lbs/hr of steam even when considerable excess water was supplied. The single conical spray nozzle was replaced with two back-to-back "fog" nozzles (Bete 1/4PJ40) operated at about 3 times the flowrate theoretically needed to desuperheat. That provided satisfactory operation up to the maximum 11.5 lb/hr of steam the generators could produce.

5.1.9 Rapid Isolation From Vacuum System

Originally, a valve in the air supply to the reboiler steam control valve was provided that allowed manual shut off of the steam as part of emergency shutdown. This valve was located near the control station to allow very rapid response. Whenever steam was suddenly shut off, the pressure would rapidly change in the crystallizer unless immediate and correct action was taken to control the bleed air to the vacuum system. After operating for a while, it was determined that rapid isolation of the crystallizer from the vacuum system was the appropriate action to be taken. The crystallizer has very little air in-leakage and is so large that the pressure does not rise appreciably for several hours. However, if the pressure decreases slightly the crystallizer contents boil violently leading to a rapid temperature decrease and subsequently further reduces the pressure. The rapid temperature drop drives the formation of many fine crystals which adversely impacts centrifuge efficiency and operations. Isolating the crystallizer from the vacuum system significantly reduces pressure decreases. An air actuated isolation valve needs to be installed between the crystallizer and the vacuum system.

5.2 Operations

5.2.1 Operating Philosophy

Operation of the FC pilot scale facility required a unique blend of conduct of operations and conduct of R&D activities. In the operations arena, the processes are well defined and well understood. The operations require a set of instructions that any operator can follow to yield the same results. In R&D world, the sole purpose of testing is often to learn the process itself while maintaining safe conditions. Thus every possible off-normal condition cannot be identified prior to testing. This requires process knowledge and operational experience to make decisions during testing. After a decision is made to resolve a given off-normal condition, step by step instructions are needed to guide the remaining shift staff. Under such conditions, it is not practical to revise the entire set of work instructions. Instead, R&D directions were written to address a specific issue. This mode of operation requires a complete and consistent understanding of system by all test engineers. Future testing will require more training on off-normal conditions and their response. For successful pilot scale operations, it has been SRNL's experience that the same people who build the system should operate it. This provides the best opportunity to learn such a complex system.

Another aspect of operations is to plan lockouts and the associated paperwork ahead of time. Often, the system lockouts are needed to resolve a technical problem immediately and any delay due to lockout preparations can further degrade the process condition.

5.2.2 Training

During the pilot scale tests it became obvious that more in-depth understanding of the system was needed for all test staff. During any unanticipated off-normal conditions, the operational instructions were not always written and ready. Hence, a thorough understanding of the process and various subsystem interactions is paramount in restoring the normal operations. Future work must be started after more training on the science behind various processes.

Similarly, more training on DAS operations will be needed before future testing. In case of unexpected shutdown of DAS or power interruptions, all test staff must have a clear understanding of how to restart DAS and create new data logs.

5.2.3 Staffing

Due to the mismatch of the centrifuge and the crystallizer and unsatisfactory performance of P&ID controllers, operations of the pilot facility became very labor intensive. Additionally, sample drawing and their preparation were also time consuming. Based upon the operational experience to date it is recommended that the following additional staff be considered for future testing:

- One more technician per shift
- An additional engineer per shift
- QEW on each shift
- Lock & Tag worker on every shift

5.2.4 Shift Turnover

The pilot scale facility was operated around the clock for several weeks by two 12-hour shifts. Each shift was comprised of a Test Engineer and a Lab Technician. At the end of every shift, a formal turnover was performed and documented by outgoing and incoming shifts. This process needs to be revised to ensure better understanding of the facility status at turnover. Since many control functions were performed manually, a detailed turnover process is needed. In particular, shift personnel coming after long breaks require more briefing about the facility status and test history during their time off.

6.0 FULL-SCALE PROCESS DESCRIPTION

The full-scale fractional crystallization system is similar to the pilot facility. However, due to the hazardous properties of the actual waste, the system is shielded and remotely operated. Process control is automatic and control systems are remote from the process.

In the full-scale system, two crystallizer systems are used. Depending upon the composition of the feed and product requirements, the crystallizer systems may be operated in series or in parallel. Parallel operation allows the highest throughput; the first stage may be used to evaporate a dilute feed to saturation prior to crystallization. Series operation allows the highest extent of cesium decontamination.

In series operation, the first crystallizer produces a rough separation of low solubility sodium nitrate, carbonate, and sulfate from high solubility cesium nitrate, sodium hydroxide, sodium aluminate, other high solubility salts (e.g. potassium hydroxide, organic salts). After product dissolution, the second crystallizer recrystallizes the low-solubility salts and decontaminates the product to the required Cs DF.

By this method, the extent of cesium decontamination is greatly increased, since the results of two-stage crystallization on Cs DF are multiplicative (e.g. Cs DF Stage 1 = 124, Stage 2 = 56, Overall Cs DF = 124 x 56 = 6,944). Overall sodium yield is similar to an one-stage crystallization, since the second stage purge is recycled to the first stage.

A process flow diagram of the proposed two-crystallizer system is shown in Figure 54. A block flow diagram and mass balances are shown in Figure 55 and 56, respectively. The mass balances are based on Group 1 IPS feed with a 1,175 MT Na/year at a 70% onstream factor. Estimated theoretical sodium yield is 67% and cesium DF is 6,937.

Figure 54. Two Stage Crystallization Process Flow Diagram

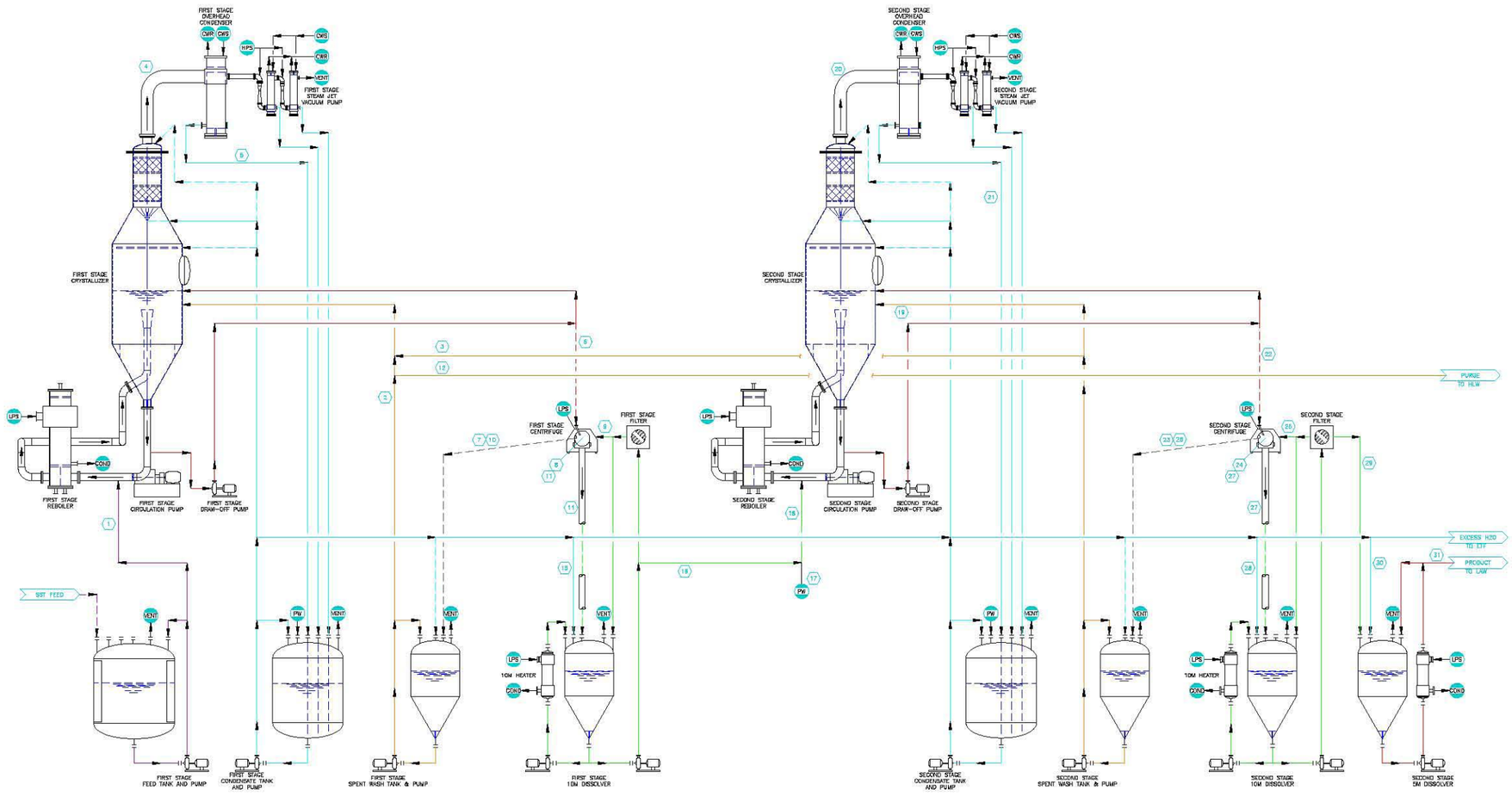


Figure 55. Two Stage Crystallization Block Flow Diagram

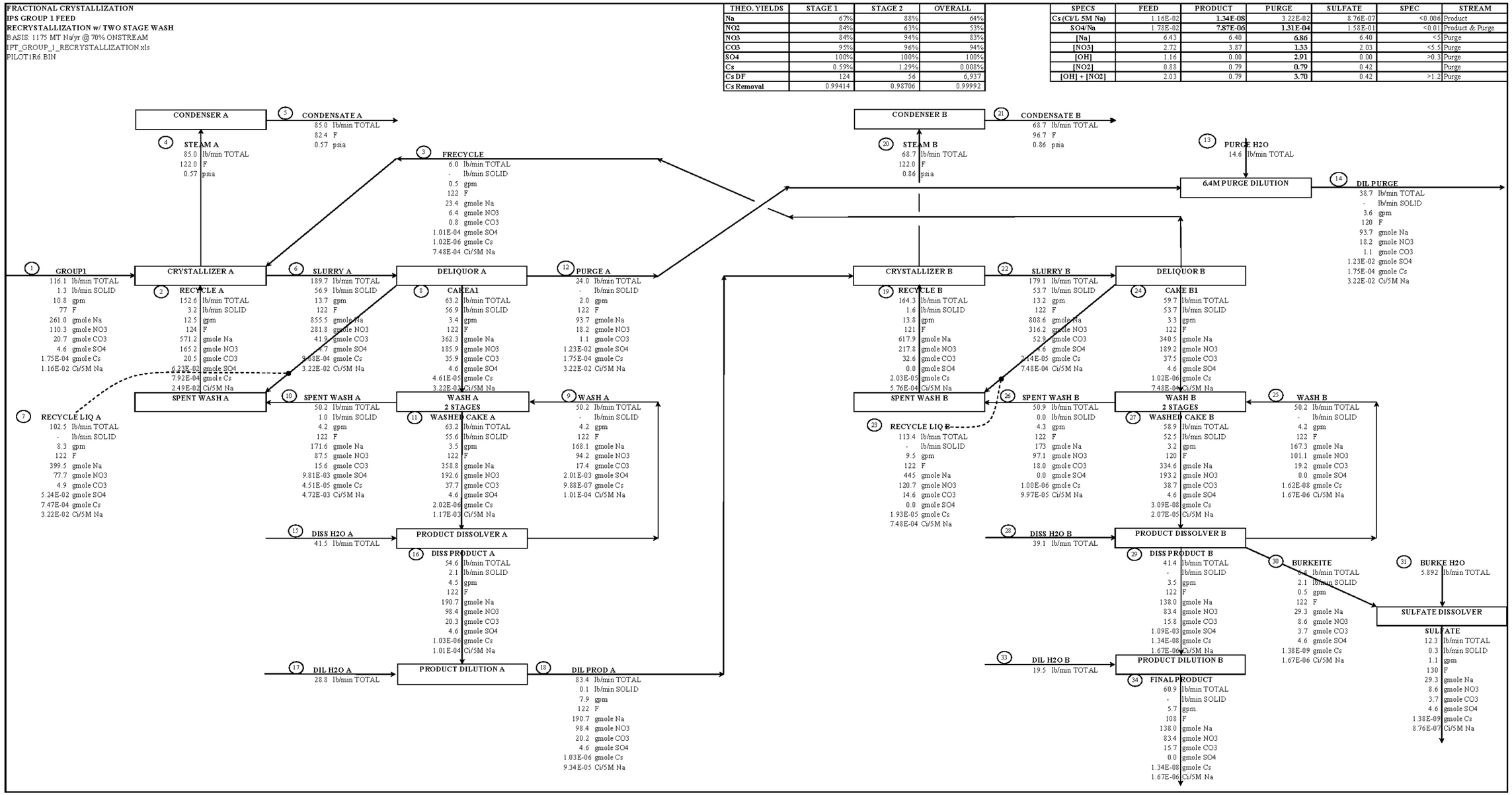


Figure 56. Two-Stage Crystallization Mass Balance

STREAM #			1		2		3	4	5	6		7	8		9	10		11	
STREAM			GROUP1	GROUP1	RECYCLE A	RECYCLE A	FRECYCLE	STEAM A	CONDENSATE A	SLURRY A	SLURRY A	RECYCLE LIQ A	CAKEA1	CAKEA1	WASH A	SPENT WASH A	SPENT WASH A	WASHED CAKE A	WASHED CAKE A
PHASE			Aqueous	Solid	Aqueous	Solid	Aqueous	Vapor	Aqueous	Aqueous	Solid	Aqueous	Aqueous	Solid	Aqueous	Aqueous	Solid	Aqueous	Solid
TEMPERATURE, F			77.0	77.0	123.9	123.9	122.0	122.0	82.4	122.0	122.0	122.0	122.0	122.0	122.0	122.0	122.0	122.0	122.0
PRESSURE, psia			14.69	14.69	14.69	14.69	14.69	0.57	0.55	0.57	0.57	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69
pH			14.42	-	15.11	-	13.84	-	6.95	15.60	-	15.60	15.60	-	14.33	13.88	-	13.97	-
COMPONENT	FORMULA	gm/gmol	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min
Water	H2O	18.02	74.02	-	61.62	-	2.18	84.97	84.97	51.90	-	40.03	2.47	-	22.33	21.83	-	2.78	-
Aluminum Hydroxide	Al(OH)3	78.00	1.07	1.22	10.32	-	0.01	-	-	12.62	-	9.74	0.60	-	0.01	0.59	-	0.03	-
Potassium Hydroxide	KOH	56.11	0.62	-	2.79	-	0.00	-	-	3.41	-	2.63	0.16	-	0.00	0.16	-	0.01	-
Sodium Hydroxide	NaOH	40.00	4.27	-	19.31	-	0.03	-	-	23.60	-	18.21	1.12	-	0.02	1.10	-	0.05	-
Sodium Nitrite	NaNO2	69.00	5.39	-	15.22	-	1.66	-	0.00	13.97	8.30	10.78	0.67	8.30	4.33	4.44	-	2.09	6.75
Sodium Nitrate	NaNO3	84.99	20.67	-	30.43	0.53	1.19	-	-	18.88	33.93	14.57	0.90	33.93	17.65	16.39	-	1.51	34.57
Sodium Carbonate	Na2CO3	105.99	4.83	-	2.49	-	0.18	-	-	1.47	-	1.13	0.07	-	4.07	2.81	-	0.22	-
Sodium Sulfate	Na2SO4	142.04	1.45	-	0.02	-	0.00	-	-	0.02	-	0.02	0.00	-	0.00	0.00	-	0.00	-
Sodium Chloride	NaCl	58.44	0.32	-	1.17	-	0.15	-	-	1.17	0.46	0.90	0.06	0.46	0.24	0.26	-	0.18	0.31
Sodium Fluoride	NaF	41.99	0.10	-	0.17	-	0.00	-	-	0.18	-	0.14	0.01	-	0.02	0.03	-	0.00	-
Sodium Orthophosphate	Na3PO4	163.94	0.24	-	1.10	-	0.00	-	-	1.34	-	1.04	0.06	-	0.00	0.06	-	0.00	-
Sodium Bicarbonate	NaHCO3	84.01	0.00	-	0.00	-	0.00	-	0.00	0.00	-	0.00	0.00	-	0.00	0.00	-	0.00	-
Sodium Chromate	Na2CrO4	161.97	0.23	-	1.03	-	0.00	-	(0.00)	1.26	-	0.97	0.06	-	0.00	0.06	-	0.00	-
Silicon Oxide	SiO2	60.08	0.03	-	0.12	-	0.00	-	-	0.15	-	0.11	0.01	-	0.00	0.01	-	0.00	-
Dawsonite	NaAlCO3(OH)2	144.00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Sodium Carbonate Monohydrate	Na2CO3.1H2O	124.00	-	-	-	2.68	-	-	-	-	9.23	-	-	9.23	-	-	0.98	-	9.54
Trisodium Fluoride Sulfate	Na3FSO4	184.03	-	-	-	0.00	-	-	-	-	0.38	-	-	0.38	-	-	-	-	0.38
Burkeite	Na6(SO4)2CO3	390.08	-	-	-	-	-	-	-	-	1.59	-	-	1.59	-	-	-	-	1.59
Sodium Acetate	Na(C2H3O2)	82.03	1.48	-	3.60	-	0.57	-	0.00	2.82	2.82	2.18	0.13	2.82	1.47	1.42	-	0.71	2.29
Sodium Oxalate	Na2C2O4	134.00	0.07	0.12	0.01	0.02	0.00	-	-	0.00	0.20	0.00	0.00	0.20	0.02	0.02	0.01	0.00	0.21
Cesium Hydroxide	CsOH	149.91	0.00	-	0.00	-	0.00	-	-	0.00	-	0.00	0.00	-	0.00	0.00	-	0.00	-
Strontium Carbonate	SrCO3	147.63	0.00	-	0.00	-	0.00	-	-	0.00	-	0.00	0.00	-	0.00	0.00	-	0.00	-
Sodium Pertechnetate	NaTcO4	185.89	0.00	-	0.00	-	0.00	-	-	0.00	-	0.00	0.00	-	0.00	0.00	-	0.00	-
Total, lb/min			114.774	1.334	149.396	3.225	5.968	84.974	84.974	132.806	56.916	102.450	6.324	56.917	50.171	49.188	0.983	7.598	55.643
Volume, gal/min			10.694	0.066	12.333	0.172	0.500	389,133.221	10.221	10.811	2.878	8.340	0.515	2.878	4.229	4.177	0.052	0.636	2.835
Enthalpy, Btu/min			-6.324E+05	-9.176E+03	-7.334E+05	-1.461E+04	-2.463E+04	-4.887E+05	-5.791E+05	-6.723E+05	-1.637E+05	-5.186E+05	-3.201E+04	-1.637E+05	-2.261E+05	-2.245E+05	-4.879E+03	-3.147E+04	-1.610E+05
Density, lb/gal			10.710	20.254	12.089	18.758	11.918	0.000	8.296	12.259	19.733	12.258	12.258	19.734	11.839	11.753	18.748	11.922	19.586
Heat Capcity, BTU/lb/F			0.684	0.277	0.592	0.280	0.645	-	0.993	0.589	-	0.590	0.590	-	0.632	0.588	0.280	0.642	0.272
Abs Visc, cP			4.092	-	6.687	-	8.356	-	0.833	8.252	-	8.252	8.252	-	6.246	5.094	-	8.333	-
Ionic Strength			7.819	-	20.770	-	21.958	-	0.000	23.639	-	23.639	23.639	-	16.276	17.324	-	21.935	-
Aqueous Sodium, molar			6.426	-	11.749	-	12.370	-	-	12.652	-	12.652	12.652	-	10.500	10.398	-	12.355	-
Total Sodium, molar			6.406	-	12.064	-	12.370	-	-	16.506	-	12.652	28.200	-	10.500	10.718	-	27.302	-
Free Hydroxide, molar			1.157	-	3.847	-	0.130	-	0.000	5.365	-	5.365	5.365	-	0.014	0.647	-	0.190	-
Soluble Alumina, molar			0.153	-	1.286	-	0.041	-	-	1.793	-	1.793	1.793	-	0.005	0.216	-	0.064	-
Total Alumina, molar			0.326	-	1.268	-	0.041	-	-	1.416	-	1.793	0.272	-	0.005	0.213	-	0.012	-
Cs Activity*, Ci/L			0.015	-	0.058	-	0.002	-	-	0.081	-	0.081	0.081	-	0.000	0.010	-	0.003	-
Cs Activity*, Ci/5M Na			0.01	-	0.02	-	0.00	-	-	0.03	-	0.03	0.03	-	0.00	0.00	-	0.00	-
Cs137/TOTAL Cs			0.30	-	0.30	-	0.30	-	0.30	0.30	-	0.30	0.30	-	0.30	0.30	-	0.30	-

STREAM #			12	13	14	15	16		17	18		19		20	21	22		23
STREAM			PURGE A	PURGE H2O	DIL PURGE	DISS H2O A	ISS PRODUCT	SS PRODUCT	DIL H2O A	DIL PROD A	DIL PROD A	RECYCLE B	RECYCLE I	STEAM B	ONDENSATI	SLURRY B	SLURRY B	RECYCLE LIQ B
PHASE			Aqueous	Aqueous	Aqueous	Aqueous	Aqueous	Solid	Aqueous	Aqueous	Solid	Aqueous	Solid	Vapor	Aqueous	Aqueous	Solid	Aqueous
TEMPERATURE, F			122.0	82.4	120.2	82.4	122.0	122.0	82.4	122.0	122.0	121.3	121.3	122.0	96.7	122.0	122.0	122.0
PRESSURE, psia			14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	0.86	0.86	0.86	0.86	14.69
pH			15.60	6.95	14.26	6.95	14.33	-	6.95	11.57	-	13.82	-	-	6.78	13.84	-	13.84
COMPONENT	FORMULA	gm/gmol	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min
Water	H2O	18.02	9.39	14.65	24.04	41.50	23.34	-	28.82	52.16	-	63.48	-	68.67	68.67	45.75	-	41.40
Aluminum Hydroxide	Al(OH)3	78.00	2.28	-	2.28	-	0.01	-	-	0.01	-	0.26	-	-	-	0.28	-	0.25
Potassium Hydroxide	KOH	56.11	0.62	-	0.62	-	0.00	-	-	0.00	-	0.07	-	-	-	0.08	-	0.07
Sodium Hydroxide	NaOH	40.00	4.27	-	4.27	-	0.03	-	-	0.03	-	0.52	-	-	-	0.55	-	0.49
Sodium Nitrite	NaNO2	69.00	2.53	0.00	2.53	0.00	4.52	-	0.00	4.52	-	35.96	-	-	0.00	34.85	5.63	31.52
Sodium Nitrate	NaNO3	84.99	3.42	-	3.42	-	18.44	-	-	18.44	-	40.34	0.48	-	-	24.99	34.26	22.61
Sodium Carbonate	Na2CO3	105.99	0.27	-	0.27	-	4.25	-	-	4.72	-	6.67	-	-	-	3.76	-	3.41
Sodium Sulfate	Na2SO4	142.04	0.00	-	0.00	-	0.00	-	-	1.45	-	0.00	-	-	-	0.00	-	0.00
Sodium Chloride	NaCl	58.44	0.21	-	0.21	-	0.25	-	-	0.25	-	3.02	-	-	-	3.06	0.21	2.77
Sodium Fluoride	NaF	41.99	0.03	-	0.03	-	0.03	-	-	0.07	-	0.08	-	-	-	0.07	-	0.06
Sodium Orthophosphate	Na3PO4	163.94	0.24	-	0.24	-	0.00	-	-	0.00	-	0.03	-	-	-	0.03	-	0.03
Sodium Bicarbonate	NaHCO3	84.01	0.00	0.00	0.00	0.00	0.00	-	0.00	0.01	-	0.00	-	-	0.00	0.00	-	0.00
Sodium Chromate	Na2CrO4	161.97	0.23	(0.00)	0.23	(0.00)	0.00	-	(0.00)	0.00	-	0.03	-	-	(0.00)	0.03	-	0.03
Silicon Oxide	SiO2	60.08	0.03	-	0.03	-	0.00	-	-	0.00	-	0.00	-	-	-	0.00	-	0.00
Dawsonite	NaAlCO3(OH)2	144.00	-	-	-	-	-	-	-	-	0.01	-	-	-	-	-	-	-
Sodium Carbonate Monohydrate	Na2CO3.1H2O	124.00	-	-	-	-	-	-	-	-	-	-	1.10	-	-	-	9.54	-
Trisodium Fluoride Sulfate	Na3FSO4	184.03	-	-	-	-	-	0.17	-	-	-	-	0.00	-	-	-	0.37	-
Burkeite	Na6(SO4)2CO3	390.08	-	-	-	-	-	1.81	-	-	-	-	-	-	-	-	1.60	-
Sodium Acetate	Na(C2H3O2)	82.03	0.51	0.00	0.51	0.00	1.53	-	0.00	1.53	-	12.25	-	-	0.00	11.87	1.91	10.74
Sodium Oxalate	Na2C2O4	134.00	0.00	-	0.00	-	0.02	0.16	-	0.13	0.05	0.03	0.01	-	-	0.02	0.21	0.01
Cesium Hydroxide	CsOH	149.91	0.00	-	0.00	-	0.00	-	-	0.00	-	0.00	-	-	-	0.00	-	0.00
Strontium Carbonate	SrCO3	147.63	0.00	-	0.00	-	0.00	-	-	0.00	-	0.00	-	-	-	0.00	-	0.00
Sodium Pertechnetate	NaTcO4	185.89	0.00	-	0.00	-	0.00	-	-	0.00	-	0.00	-	-	-	0.00	-	0.00
Total, lb/min			24.031	14.650	38.682	41.503	52.432	2.141	28.822	83.336	0.059	162.749	1.580	68.669	68.667	125.341	53.717	113.393
Volume, gal/min			1.956	1.762	3.611	4.992	4.420	0.100	3.467	7.854	0.003	13.703	0.084	##########	8.281	10.495	2.751	9.494
Enthalpy, Btu/min			-1.217E+05	-9.985E+04	-2.215E+05	-2.829E+05	-2.363E+05	-9.182E+03	-1.964E+05	-4.406E+05	-2.616E+02	-6.893E+05	-6.581E+03	-3.949E+05	-4.670E+05	-5.173E+05	-1.560E+05	-4.680E+05
Density, lb/gal			12.258	8.297	10.691	8.297	11.839	21.329	8.297	10.589	19.584	11.853	18.764	0.000	8.275	11.918	19.489	11.918
Heat Capcity, BTU/lb/F			0.590	0.993	0.691	0.993	0.632	0.226	0.993	0.728	0.208	0.638	0.280	-	0.995	0.645	-	0.645
Abs Visc, cP			8.252	0.833	3.121	0.833	6.246	-	0.833	2.451	-	7.543	-	-	0.707	8.353	-	8.356
Ionic Strength			23.639	0.000	9.192	0.000	16.276	-	0.000	7.854	-	20.081	-	-	0.000	21.960	-	21.958
Aqueous Sodium, molar			12.652	-	6.855	-	10.500	-	-	6.400	-	11.706	-	-	-	12.371	-	12.370
Total Sodium, molar			12.652	-	6.855	-	11.142	-	-	6.410	-	11.838	-	-	-	16.124	-	12.370
Free Hydroxide, molar			5.365	0.000	2.909	0.000	0.014	-	0.000	0.012	-	0.094	-	-	0.000	0.130	-	0.130
Soluble Alumina, molar			1.793	-	0.971	-	0.005	-	-	0.002	-	0.030	-	-	-	0.041	-	0.041
Total Alumina, molar			1.793	-	0.971	-	0.005	-	-	0.003	-	0.030	-	-	-	0.032	-	0.041
Cs Activity**, Ci/L			0.081	-	0.044	-	0.000	-	-	0.000	-	0.001	-	-	-	0.002	-	0.002
Cs Activity**, Ci/5M Na			0.03	-	0.03	-	0.00	-	-	0.00	-	0.00	-	-	-	0.00	-	0.00
Cs137/TOTAL Cs			0.30	0.30	0.30	0.30	0.30	-	0.30	0.30	-	0.30	-	-	0.30	0.30	-	0.30

STREAM #			24		25	26		27		28	29	30		31	32		33	34	35
STREAM			CAKE B1	CAKE B1	WASH B	SPENT WASH B	PENT WASH	ASHED CAKE	WASHED CAKE B	DISS H2O B	SS PRODUCT	BURKEITE	BURKEITE	BURKE H2O	SULFATE	SULFATE	DIL H2O B	FINAL PRODUCT	EXCESS H2O
PHASE			Aqueous	Solid	Aqueous	Aqueous	Solid	Aqueous	Solid	Aqueous	Aqueous	Aqueous	Solid	Aqueous	Aqueous	Solid	Aqueous	Aqueous	Aqueous
TEMPERATURE, F			122.0	122.0	122.0	122.0	122.0	120.0	120.0	96.6	122.0	122.0	122.0	96.6	129.9	129.9	96.6	108.2	96.6
PRESSURE, psia			14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69	14.69
pH			13.84	-	13.09	14.73	-	19.29	-	6.77	13.09	13.09	-	6.77	10.96	-	6.77	11.37	6.77
COMPONENT	FORMULA	gm/gmol	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min	lb/min
Water	H2O	18.02	2.18	-	22.38	22.25	-	2.29	-	39.05	18.46	1.91	-	5.89	7.80	-	19.50	37.96	4.22
Aluminum Hydroxide	Al(OH)3	78.00	0.01	-	0.00	0.01	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Potassium Hydroxide	KOH	56.11	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Sodium Hydroxide	NaOH	40.00	0.03	-	0.00	0.03	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.01	-
Sodium Nitrite	NaNO2	69.00	1.66	5.64	3.14	4.44	-	1.77	4.23	0.00	2.59	0.27	-	0.00	0.27	-	0.00	2.59	0.00
Sodium Nitrate	NaNO3	84.99	1.19	34.26	18.95	18.20	-	1.24	34.96	-	15.63	1.62	-	-	1.62	-	-	15.63	-
Sodium Carbonate	Na2CO3	105.99	0.18	-	4.48	4.20	-	0.36	-	-	3.69	0.38	-	-	0.87	-	-	3.67	-
Sodium Sulfate	Na2SO4	142.04	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	1.33	-	-	0.00	-
Sodium Chloride	NaCl	58.44	0.15	0.20	0.12	0.25	-	0.15	0.07	-	0.10	0.01	-	-	0.01	-	-	0.10	-
Sodium Fluoride	NaF	41.99	0.00	-	0.02	0.02	-	0.00	-	-	0.02	0.00	-	-	0.01	-	-	0.02	-
Sodium Orthophosphate	Na3PO4	163.94	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Sodium Bicarbonate	NaHCO3	84.01	0.00	-	0.00	0.00	-	0.00	-	0.00	0.00	0.00	-	0.00	0.00	-	0.00	0.02	0.00
Sodium Chromate	Na2CrO4	161.97	0.00	-	0.00	0.00	-	0.00	-	(0.00)	0.00	0.00	-	(0.00)	0.00	-	(0.00)	0.00	(0.00)
Silicon Oxide	SiO2	60.08	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Dawsonite	NaAlCO3(OH)2	144.00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Sodium Carbonate Monohydrate	Na2CO3.1H2O	124.00	-	9.54	-	-	-	-	9.65	-	-	-	-	-	-	-	-	-	-
Trisodium Fluoride Sulfate	Na3FSO4	184.03	-	0.37	-	-	-	-	0.37	-	-	-	0.18	-	-	0.15	-	-	-
Burkeite	Na6(SO4)2CO3	390.08	-	1.60	-	-	0.00	-	1.60	-	-	-	1.81	-	-	-	-	-	-
Sodium Acetate	Na(C2H3O2)	82.03	0.57	1.91	1.06	1.50	-	0.59	1.44	0.00	0.88	0.09	-	0.00	0.09	-	0.00	0.88	0.00
Sodium Oxalate	Na2C2O4	134.00	0.00	0.21	0.03	0.02	0.00	0.00	0.21	-	0.02	0.00	0.16	-	0.02	0.14	-	0.02	-
Cesium Hydroxide	CsOH	149.91	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Strontium Carbonate	SrCO3	147.63	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Sodium Pertechnetate	NaTcO4	185.89	0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Total, lb/min			5.970	53.728	50.171	50.934	0.002	6.404	52.529	39.053	41.391	4.282	2.141	5.892	12.022	0.293	19.500	60.892	4.224
Volume, gal/min			0.500	2.751	4.208	4.275	0.000	0.527	2.709	4.709	3.472	0.359	0.100	0.710	1.122	0.014	2.351	5.696	0.509
Enthalpy, Btu/min			-2.464E+04	-1.561E+05	-2.267E+05	-2.279E+05	-7.784E+00	-2.646E+04	-1.530E+05	-2.656E+05	-1.870E+05	-1.935E+04	-9.185E+03	-4.007E+04	-6.731E+04	-1.295E+03	-1.326E+05	-3.196E+05	-2.873E+04
Density, lb/gal			11.918	19.489	11.897	11.890	19.680	12.118	19.350	8.276	11.897	11.897	21.330	8.276	10.697	20.894	8.276	10.668	8.276
Heat Capcity, BTU/lb/F			0.645	-	0.624	0.627	0.230	0.653	0.274	0.994	0.624	0.624	-	0.994	0.701	0.230	0.994	0.730	0.994
Abs Visc, cP			8.356	-	6.140	6.480	-	10.271	-	0.707	6.140	6.140	-	0.707	1.727	-	0.707	2.694	0.707
Ionic Strength			21.958	-	16.227	16.866	-	22.345	-	0.000	16.227	16.227	-	0.000	7.186	-	0.000	7.923	0.000
Aqueous Sodium, molar			12.370	-	10.500	10.703	-	12.914	-	-	10.500	10.500	-	-	6.400	-	-	6.400	-
Total Sodium, molar			27.666	-	10.500	10.704	-	27.304	-	-	10.500	16.823	-	-	6.805	-	-	6.400	-
Free Hydroxide, molar			0.130	-	0.000	0.015	-	0.004	-	0.000	0.000	0.000	-	0.000	0.005	-	0.000	0.005	0.000
Soluble Alumina, molar			0.041	-	0.000	0.005	-	0.001	-	-	0.000	0.000	-	-	0.000	-	-	0.000	-
Total Alumina, molar			0.006	-	0.000	0.005	-	0.000	-	-	0.000	0.000	-	-	0.000	-	-	0.000	-
Cs Activity*, Ci/L			0.002	-	0.000	0.000	-	0.000	-	-	0.000	0.000	-	-	0.000	-	-	0.000	-
Cs Activity*, Ci/5M Na			0.00	-	0.00	0.00	-	0.00	-	-	0.00	0.00	-	-	0.00	-	-	0.00	-
Cs137/TOTAL Cs			0.30	-	0.30	0.30	-	0.30	-	0.30	0.30	0.30	-	0.30	0.30	-	0.30	0.30	0.30

Tank waste is first filtered to remove suspended solids. Filtrate flows to the Feed Receipt Tank, which provides buffer storage capacity. Waste feed is transferred continuously to the first stage crystallizer to maintain stable steady-state operating conditions.

The crystallizer is operated under vacuum (typically .035 to 0.1 atmospheres absolute) to maintain boiling temperatures in the 40 to 60ºC range needed for crystallization. A relatively large recirculation stream flows from the bottom of the crystallizer through the steam-heated reboiler, which provides heat for evaporation. The crystallizer is sized for an eight-hour residence time.

Steam from the crystallizer flows through a demister in the top of the crystallizer to remove droplets and aerosols, and then flows to the first stage condenser where the bulk of the water vapor is condensed. Remaining water vapor and non-condensable gases then flow through two steam jet eductors with condensers that maintain vacuum on the crystallizer. Pressure on the crystallizer is maintained by control of purge air into the steam jet system. Non-condensables are filtered prior to discharge to the environment

Process condensate is collected from the primary condenser and steam jets and is used for dissolution and dilution of product as needed. Surplus process condensate is transferred to an external treatment facility, assumed to be the Effluent Treatment Facility (ETF). Efficient de-entrainment is required to remove waste particles from steam generated in the crystallizer. To assure ETF acceptance requirements for ^{137}Cs are met a minimum decontamination factor of 3×10^{5} is specified as a design requirement for the crystallizer overhead steam system.

Slurry containing crystals and liquor is drawn off the crystallizer and pumped to a centrifuge for deliquoring and decontamination. The slurry is initially deliquored to 90% solids by centrifugal force in the centrifuge. Then a recycled product stream (10 M Na) is sprayed on the centrifuge cake to displace interstitial contamination.

The centrifuge cake is discharged to a product tank where dissolution, heating, and filtration occur. The product is dissolved to 10M Na to produce liquor that is saturated in sodium nitrate for recycle washing of the filter cake. A heater maintains the heat during endothermic dissolution of sodium nitrate. The liquor is oversaturated in sodium sulfate and carbonate. A filter removes the suspended solids from the recycle to the centrifuge.

A portion of the spent liquor stream discharged from the centrifuge is recycled to the crystallizer, while the remainder is purged to the Cesium Product Tank. The purge is diluted with water to 6M Na in the Cesium Product Tank prior to return to a DST to prevent crystallization upon cooling.

The second stage crystallizer system operates essentially the same as the first stage, except that feed comes from the first stage dissolver and the purge is recycled to the first stage crystallizer. The second stage dissolver and product filter separates undissolved sulfate from the LAW product. Sodium concentration is controlled at 10 M Na so that the sulfate remains as undissolved crystals. It is separated from the bulk of the LAW product by filtration. The high-sulfate stream from the filter is collected in a separate High Sulfate

Product Tank and may be split between the LAW Product Tank and the Cesium Product Tank in order to control the amount of sulfate in the LAW product. The combined LAW products are accumulated in the LAW Product Tanks and the final product is further diluted with water to dissolve remaining crystals prior to transfer to WTP.

Because the first stage delivers a concentrated product, the second-stage crystallizer evaporation duty is lower than the first stage. Operating conditions and stream properties in the second stage may also be different because of the reduced concentration of high solubility waste components such as sodium and potassium hydroxide and sodium aluminate. The absence of these components in the second stage allows a higher extent of evaporation and a higher sodium yield (88 vs. 67%) than the first stage. As a result, the second stage requires a higher liquor recycle rate from the centrifuge to maintain the slurry solids fraction <30%. At the same operating temperature, the second crystallizer has higher vapor pressure due to the lack of high-solubility salts.

The process is controlled to maintain steady-state operation of the entire crystallizer system. Process variables, including temperatures, pressures, crystallizer slurry density, flow rates, tank levels, etc. are measured and controlled to maintain process variables at set point values. The crystallizer level is maintained constant by feed makeup. The crystallizer temperature is controlled by vacuum control in the overhead system. The evaporation rate is controlled by the steaming rate in the reboiler. The slurry density and solids fraction are maintained by the centrifuge production rate and spent liquor recycle rate.

Routine sampling and analysis is not expected to be needed for process control, assuming the feed has been characterized in advance for each batch. Sampling of selected process streams is performed occasionally on an as-needed basis to support optimization, troubleshooting, and regulatory compliance documentation. Efficiency of ^{137}Cs decontamination in each crystallizer stage is monitored in real time by measuring gamma radiation dose rates of the dissolved product. Amount of dissolved product recycled for washing centrifuge cake is adjusted as needed to assure target ^{137}Cs decontamination is achieved. Off-specification product is diluted and recycled to the crystallizer feed tank.

Process lines containing slurry are heat traced and insulated to prevent cold spots where pluggage may occur. Draining and flushing capability is provided to prevent line pluggage, reduce personnel dose rate, and allow certain maintenance functions to be performed. Capability is provided to empty the crystallizer contents to the feed tank during unplanned shutdowns.

6.1 Full-Scale Equipment List

The following equipment list and facility layouts are excerpted from report RPP-RPT-37551, 2008, *Project W-551 Interim Pretreatment System Pre-conceptual Candidate Technology Descriptions.* The equipment sizing was based the flowsheet and mass balances from the previous section. Design capacity was based on Group 1 IPS feed with a 1,175 MT Na/year at a 70% onstream factor.

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Feed Receipt Tank	32,000 gal total capacity	17.6-ft D x 17.6-ft H	Clean out jet to empty tank in case of failed pump Nozzles: (3) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Provides 1 day of crystallizer feed for the worst case feed flow rate
1	Feed Pump, Crystallizer 1	25 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate is the maximum waste feed flow rate of 17.3 gpm.

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
2	LAW Product Tanks	23,000 gal total capacity	15.8-ft D x 15.8-ft H	Clean out jet to empty tank in case of failed pump Nozzles: (4) process piping (1) off gas (1) pump (1) mixer (3) instrumentation (1) sample (1) PRV I&C: Level Temperature Pressure control (offgas) Radiation monitor on inflow	Working volume for storing four days of treated LAW at the maximum production rate in each tank
2	LAW Product Pumps	100 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Design basis flow rate for feed to LAW is 88 gpm.
1	Cs Product Tank	8,600 gal total capacity	11.4-ft D x 11.4-ft H	Clean out jet to empty tank in case of failed pump Nozzles: (4) process piping (1) off gas (1) pump (3) instrumentation (1) sample (1) PRV I&C: Level Temperature control Pressure control (offgas)	Volume for storing one day of input from the worst case throughput.

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Cs Product Pump	100 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate for return to tank farms assumed to be approximately 75 gpm.
	1st Stage Crystallization				
1	Reboiler, 1st stage	11,000,000 BTU/hr		Includes small condensate pump	Sized based on highest boilup rate.
1	Crystallizer, 1st stage	6200 gal working volume	8 ft minimum freeboard including transition to demister. Approx 9 ft 9 inch outside diameter X 23 ft tall vessel plus 4ft dia X 4 ft demister section on top.	Design for full vacuum	Crystallizer volume is sized to provide an 8 hour residence time at the maximum centrifuge feed rate (Stream 19).
1	Crystallizer Recirculation Pump, 1st stage	5600 gpm low head 20 psig 125 HP			Flow based on scale factor comparing the boilup rate with that of the 242A Evaporator (0.4 for Stage 1).
1	Condenser, 1st stage	8,500,000 Btu/hr		Includes small condensate pump	Sized to condense maximum boilup rate.
1	Condenser Vacuum Pump (steam powered eductor), 1st stage	131 lb/hr saturated air, 0.035 atm suction pressure		Steam Powered Eductor	Non-condensable suction flow of 40 lb/hr based on scale factor comparing the boilup rate with that of the 242A Evaporator (0.4 for Stage 1).
1	Centrifuge Feed Pump, 1st stage	15 gpm crystal slurry, density 1.74			Based on maximum steady state flow of slurry from the crystallizer (Stream 19)
1	Centrifuge, 1st stage	15 gpm crystal slurry, feed, 1800 kg/hr (4000 lb/hr) solids product		Peeler type centrifuge	Based on maximum steady state flow of slurry from the crystallizer (Stream 19)

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Dissolver Tank, 1st stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (4) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank
1	Dissolver Recirculation Pump , 1st stage	60 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Sized for maximum heat exchanger temperature difference of 20F.
1	Dissolver Discharge Pump , 1st stage	20 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 1.5X the worst case dissolver discharge flow rate.
1	Dissolver filter, 1st stage	Feed 15 gpm, filtrate 6 gpm		5 micron pore size, 70 psi filter differential pressure	Filtrate is 1.5 X maximum steady state rate (Batch 2)
1	Dissolver Heat Exchanger, 1st stage	600,000 Btu/hr			Sized to heat highest flow rate of added dissolution water from 70 F to 140 F, times 2X.
1	Condensate Tank, 1st stage	2,000 gal working volume	7.0-ft D x 7.0-ft H	Outside, double wall tank Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas) Leak detection	Volume to store 2 hour capacity for worst case condensate flow rate.

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Condensate Pump, 1st stage	40 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 2.5X the worst case condensate flow rate.
1	Spent Wash Tank, 1st stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank
1	Spent Wash Pump, 1st stage	15 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 2.5X the worst case wash liquor flow rate.
1	Centrifuge Liquor Tank, 1st stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank
1	Centrifuge Liquor pump, 1st stage	25 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 2.5X the worst case crystallizer product flow rate.
	2nd Stage Crystallization				

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Reboiler, 2nd stage	5,500,000 BTU/hr		Includes small condensate pump	Sized based on highest boilup rate.
1	Crystallizer, 2nd stage	6200 gal working volume	8 ft minimum freeboard including transition to demister. Approx 9 ft 9 inch outside diameter X 23 ft tall vessel plus 4ft dia X 4 ft demister section on top.	Design for full vacuum	Crystallizer volume is sized to provide an 8 hour residence time at the maximum centrifuge feed rate (Stream 19). 5000 gal for Stage 2, but Crystallizer is sized to be equal to Stage 1.
1	Crystallizer Recirculation Pump, 2nd stage	2800 gpm low head 20 psig 75 HP			Flow based on scale factor comparing the boilup rate with that of the 242A Evaporator (0.2 for Stage 2).
1	Condenser, 2nd stage	4,500,000 Btu/hr		Includes small condensate pump	Sized to condense maximum boilup rate.
1	Condenser Vacuum Pump (Steam Powered Eductor), 2nd stage	65 lb/hr air saturated with water vapor, 0.57 psia suction pressure		(Steam Powered Eductor)	Non-condensable suction flow of 20 lb/hr based on scale factor comparing the boilup rate with that of the 242A Evaporator (0.2 for Stage 2).
1	Centrifuge Feed Pump, 2nd stage	Size same as first stage			Size same as first stage
1	Centrifuge, 2nd stage	Size same as first stage		Peeler type centrifuge	Size same as first stage
1	Dissolver Tank, 2nd stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (4) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank
1	Dissolver Recirculation Pump , 2nd stage	30 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Sized for maximum heat exchanger temperature difference of 20F.

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Dissolver Discharge Pump , 2nd stage	15 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 1.5X the worst case dissolver discharge flow rate.
1	Dissolver Heat Exchanger, 2nd stage	300,000 Btu/hr			Sized to heat highest flow rate of added dissolution water from 40 F to 140 F, times 2X.
1	Dissolver filter, 2nd stage	Feed 10 gpm, filtrate 8 gpm		5 micron pore size, 70 psi filter differential pressure	Feed is 1.5 X and filtrate is 1.4 X maximum steady state rate for filter feed (Batch 2). Sized to allow flexibility to provide concentrated sulfate product (filter product slurry)
1	Condensate Tank, 2nd stage	2,000 gal total capacity	7.0-ft D x 7.0-ft H	Outside, double wall tank Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas) Leak detection	Volume to store 2 hour capacity for worst case condensate flow rate.
1	Condensate Pump, 2nd stage	20 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 2.5X the worst case condensate flow rate.

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	Spent Wash Tank, 2nd stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank
1	Spent Wash Pump, 2nd stage	10 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Flow rate based on 2.5X the worst case wash liquor flow rate.
1	Centrifuge Liquor Tank, 2nd stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank.
1	Centrifuge Liquor pump, 2nd stage	20 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	2.5 times maximum Stream 31 liquid phase

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
1	High sulfate product Tank, 2nd stage	1,000 gal working volume	5.0-ft D x 10.0-ft H	Nozzles: (2) process piping (1) off gas (1) pump (3) instrumentation (1) PRV I&C: Level Temperature Pressure control (offgas)	Standardized in-cell process tank
1	High sulfate product Pump, 2nd stage	10 gpm		Tank top mounted, vertical pump I&C: Discharge pressure Flow control VFD	Sized the same as the 2nd stage dissolver pump
	Miscellaneous				
1	Steam Supply System	25,000 lb/hr 100 psig (vacuum pumps) 3 psig (reboilers)		Loads: (2) Reboilers (2) Vacuum Pumps (2) Dissolver Hx	Based on condensing 3 psig steam in reboiler and dissolver plus vacuum pump steam demand.
	Structures, FC System				
1	Crystallizer Building		80' L x 30' W x 38' H Base 18' below grade	Separate process, maintenance and operating areas. See layout. Process area below grade with similar construction as Tank/Equipment vaults.	See section 8 for description of layout

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments
4	Tank/Equipment Vaults Feed Receipt Tank Cs Product Tank LAW Product Tank #1 LAW Product Tank #2		22' L x 22' W x 25' H 22' L x 16' W x 25' H 23' L x 20' W x 25' H 23' L x 20' W x 25' H Internal Dimensions	Concrete below grade structure with 3-ft thick walls and floors 3-ft thick concrete cover blocks at grade consisting of 12" wide removable concrete beams. Stainless steel lined floor and walls up to bottom of cover blocks Sump with remote read-out leak detector and sump pump for each vault Remote connector heads	
1	Valve Vault		22' L x 10' W x 15' H Internal Dimensions	One valve vault adjacent to and serving all tank vaults Concrete below grade structure with 3-ft thick walls and floors 3-ft thick concrete cover blocks at grade consisting of 12" wide removable concrete beams. Stainless steel lined floor and walls up to bottom of cover blocks Sump with remote read-out leak detector and sump pump Remote connector heads	
4	Equipment Pads Condensate Tank, 1st Stage Condensate Tank, 2nd Stage Steam Boiler Chiller (see Common Equipment)		17' L x 17' W 14' L x 14' W 16' L x 14' W	8-in thick pad	

Table 12. Fractional Crystallization Equipment List
(11 sheets)

Qty	Component	Process Sizing	Physical Dimensions	Features	Comments

General Notes:

1. All tanks are designed, fabricated and tested to ASME Section VIII
2. All process piping is designed, fabricated and tested to ASME B31.3
3. All process equipment, chemical equipment and offgas piping are manufactured from 304L or 316L SS.
4. See Common Equipment List for process offgas, vault ventilation, recirculation AHU, and chilled water systems.
5. Tanks are sized assuming a working volume equal to 80% of the total capacity.

Figure 57. Full-Scale Equipment Layout

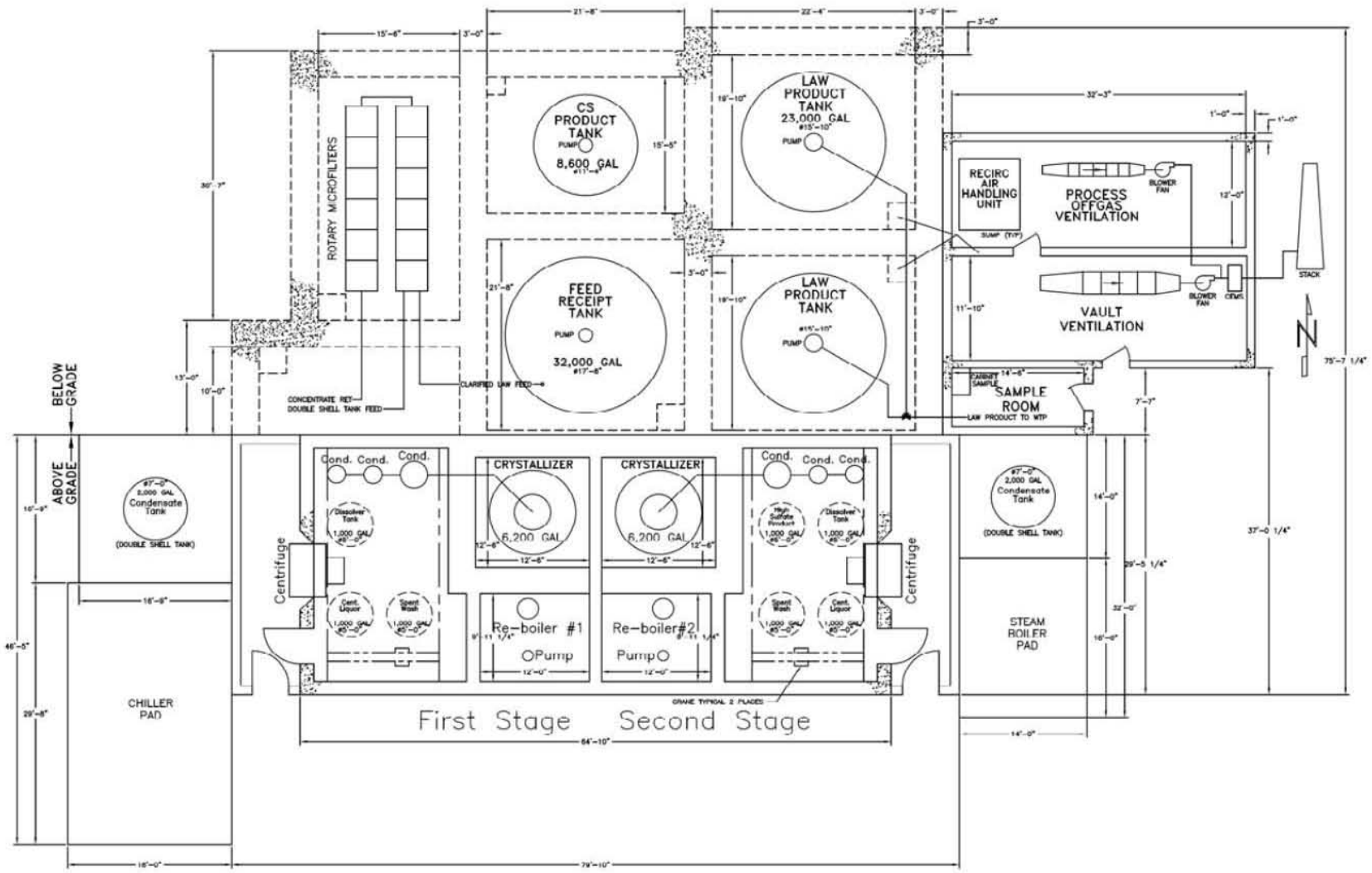
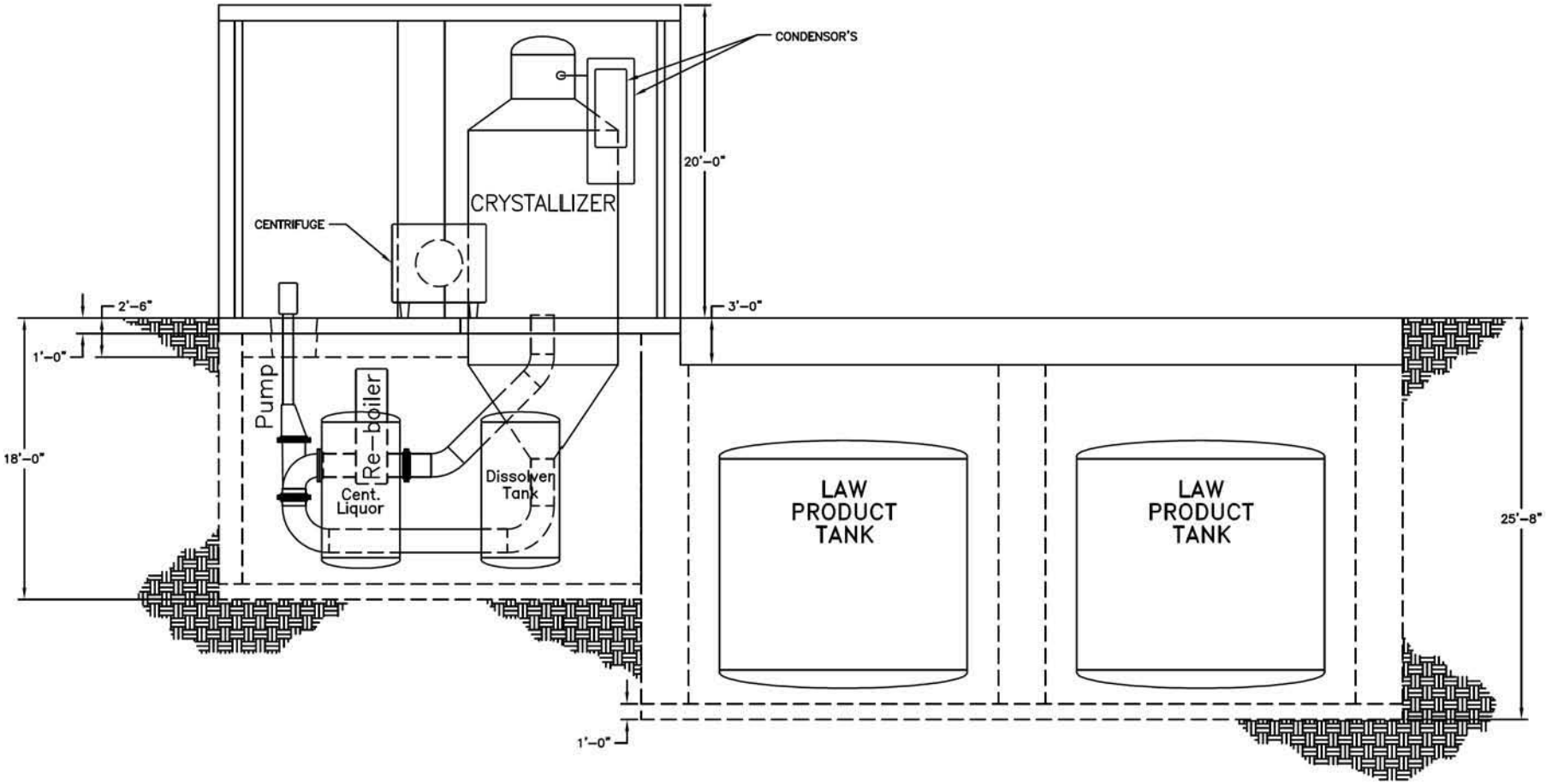


Figure 58. Full-Scale Equipment Elevation View



7.0 CONCLUSIONS

As noted in Section 3.0, two important goals were set for the FCPP operations: 1) resolve the ITDP enabling assumptions and uncertainties and 2) meet contract process performance requirements.

7.1 ITDP Enabling Assumptions

The ITDP enabling assumptions were used as the basis for continuing project activities before results could demonstrate they were indeed factual.

7.1.1 Modeling – Enabling Assumption

"Modeling can be used to predict FC process performance with Hanford Waste" The same thermodynamic modeling program was used to accurately predict product yield and DF results for all laboratory, engineering, and pilot scale testing activities. Confirmation that the simulant and actual waste behaved in the same manner is documented in RPP-RPT-31352, *Fractional Crystallization Flowsheet Tests with Actual Tank Waste.* In addition, the model was used to guide simulant testing using different laboratory crystallizers, i.e. the Hanford Boildown Apparatus (HBA) vs. the Georgia Tech jacketed and stirred crystallizer, with both units producing similar results as reported in RPP-RPT-30905, *Fractional Crystallization Simulant Test Comparisons.*. Therefore since the same model was used and results did not differ between actual waste and simulant, or with the type of test apparatus, the enabling assumption is proved "true". The model was also used for the FCPP and was the starting point to establish the pilot system operating parameters.

7.1.2 Simulant Performance – Enabling Assumption

"Simulant can be used to investigate FC performance with actual Hanford Waste." Early in Phase II of the project the simulant and actual waste were tested "side-by-side" at the bench scale and found to yield similar results as noted in RPP-RPT-31352, *Fractional Crystallization Flowsheet Tests with Actual Tank Waste*. The laboratory simulant formulation (at a larger scale) was used in the pilot and behaved as it did during all bench top testing therefore the enabling assumption is proved "true".

7.1.3 Scalability – Enabling Assumption

"Fractional Crystallization is scalable; specifically lab scale, engineering scale, and pilot scale tests can be used to investigate full scale process performance." As noted in the reports referenced above, the simulant acted like the real waste and different laboratory crystallizers using the same simulant produced similar results. Testing at the Swenson development Laboratory in Harvey, IL utilized a 20 liter (5 gallon), continuous flow, draft tube entry (DTE) crystallizer as opposed to the 150 mL, 250 mL, 600 mL, and 1 L semi-batch crystallizers used in bench top testing. In addition, the Swenson tests used a centrifuge for solid-liquid separation instead of the Büchner funnel (vacuum assisted) apparatus used in the laboratory. Results demonstrated that crystal morphology remained the same, but dewatering of the cake improved dramatically with the centrifuge. Details of

the testing and results are contained in RPP-RPT-33228, *Hanford medium/Low Curie Waste Pretreatment Alternatives Project – Phase II report on Pre-Pilot Work at Swenson Technology, Inc.* In the pilot crystallizer volume increased to about 1200 gallons and crystal morphology was controlled to yield results similar to laboratory results as verified by PLM and CSD analyses. There fore the enabling assumption is proved "true."

7.2 Uncertainties

Even though crystallization with product removal is a common industrial process, it was new to normal Hanford operations so uncertainties arose concerning how the FCPP (and a potential production unit) would operate. The PTP and Analytical Test Plan were developed to address these uncertainties.

7.2.1 Flexibility Uncertainty

"Ability to handle feed variability including; solids, organics and recycle streams." Due to the curtailed testing program, the effects of high solids or organics content in the feed stream could not be tested in the FCPP. Recycling of the product wash stream within the crystallizer circuit had no adverse effects and was in fact necessary to maintain balanced chemistry conditions within the unit. This uncertainty, in respect to high solids and organics, remains "open."

7.2.2 Efficiency Uncertainty

"Efficiency of separating salt crystals from mother liquor containing Cs, Tc, and I". Only non-radioactive Cs was added to the simulant to determine the efficiency (as indicated by DF results) of the solid-liquid separation stage. In the proposed operating plant DF would normally be measured by analyzing the dissolved product, but because the product tank was contaminated with mother liquor (containing the non-radioactive Cs) DF analysis was performed on cake samples taken directly from the centrifuge discharge chute. The contractual requirement for product DF was 50 and even with the process upsets encountered during FCPP operations, final DFs ranged from 100 to 200. Therefore pilot plant results indicate that Cs (and by extension Tc and I based on RPP-RPT-31352) can be effectively separated from solid product.

7.2.3 Crystal Growth Uncertainty

"Crystallizer retention time required for crystal growth". Several crystal species, e.g. sodium nitrate, sodium carbonate, burkeite, etc., nucleate concurrently in the simulant and need time to mature (grow) before harvesting in the centrifuge. Bench-top and engineering scale experiments indicated that the optimum size range for the predominant crystal species (sodium nitrate) should be 300 to 450 μ. Pilot results (by PLM and CSD analyses) indicate that this size range was easily achievable with the inlet extension pieces removed (yielding minimum retention time). Therefore the retention time is adequate.

7.2.4 Complexity of Control System Uncertainty

"Complexity of control system, ease of operation." The pilot system contained several instruments and controls for the purpose of gathering operational data "in case" it could be used for later evaluation. Some of the process control functions were never able to be properly adjusted e.g. crystallizer pressure control, crystallizer level control, density controller on the Product Dissolver Tank, reboiler steam flow control, speed control on the draw-off loop, etc. so that the entire system had to be controlled in "manual" mode. The centrifuge PLC operated satisfactorily (feed, spin, wash, and peel), but since an automatic speed control had not been purchased for the hydraulic circuit, manual speed changes were necessary for heel removal. Manual operation demonstrated that only a few key variables need to be manipulated to achieve satisfactory results.

7.2.5 Robustness Uncertainty

"Robustness to Process Upset." As noted above, automatic operation of the system was not achievable. This resulted in many cases of operator error causing process upsets in addition to other, sometimes concurrent, unplanned events like power outages, pipe breaks, pump failures, pipe plugging, etc. Throughout these events corrective actions were implemented that eventually returned the system to stable operation. Since the system was able to meet process requirements in spite of many upset conditions, robustness is not an issue.

7.3 Summary

Summarizing the above, the FCPP has:

- Achieved an Average Filter Cake Cesium Decontamination Factor of 130, compared to a goal of at least 50.
- Achieved a Sodium Product Yield (percentage of sodium isolated to send to LAW vitrification) of 52%, compared to a goal of 50%.

The pilot system effectively operated with a varying range of operating parameters, e.g. crystallizer temperature, pressure and level, slurry density, product dissolver density, etc.

Operators were able to rapidly recover the system after major upsets, e.g. loss of steam, pipe breaks, line pluggage, loss of operating instrumentation, etc.

The thermodynamic model was again shown to be able to closely predict actual results within plant limitations.

Failure of key instruments (crystallizer pressure and level control and dissolver tank density control) and equipment (dissolver tank cross-flow filter) limited the performance of the fractional crystallization pilot system.

7.4 Future Work

FC has been shown to be an effective pretreatment method for SST wastes. Completing the original scope of testing and making a few system modifications (based on recent operating observations) could erase any questions not answered by the current pilot plant data. Extrapolating the pilot data and combining them with results from recent DST simulant testing at 222-SA (see CH2M HILL Interoffice Memorandum 74A10-DLH-08-155) suggests that certain DST wastes could be processed effectively by FC with little or no modification to the basic process design. Before processing any new feed for the FC process, thermodynamic modeling should be performed to determine potential product solubilities and operating parameters.

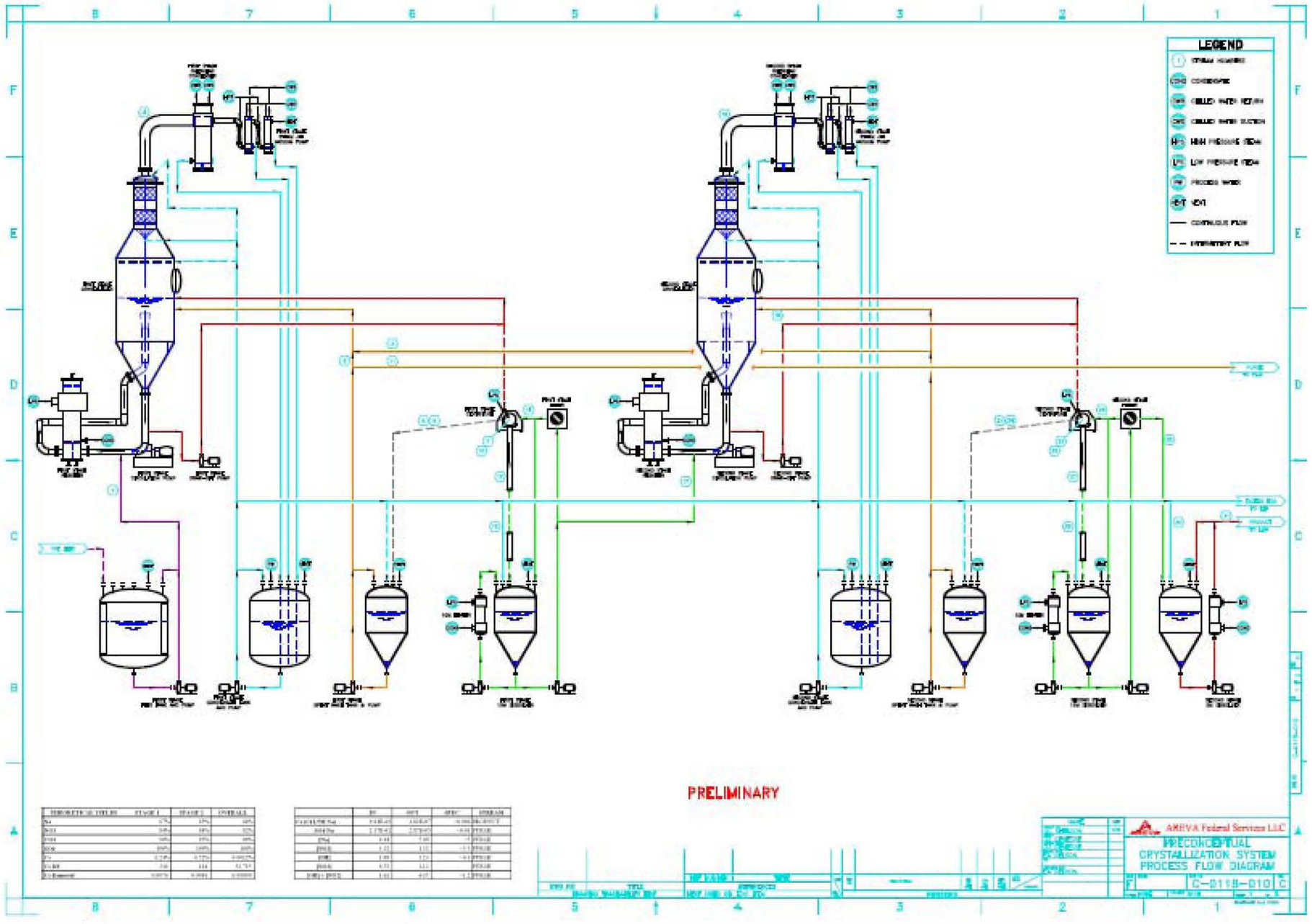
8.0 APPENDICES

Appendix 8.1 - Process Flow Diagram
Appendix 8.2 - Process and Instrumentation Diagram
Appendix 8.3 - Northwest Copper Crystallizer Drawing
Appendix 8.4 - Northwest Copper Reboiler Drawing
Appendix 8.5 - Pilot Instrument List
Appendix 8.6 - 8.6 Pilot Valve List
Appendix 8.7 - Pilot DAS Indications
Appendix 8.8 - Pilot Equipment List
Appendix 8.9 - Readiness Certification Document
Appendix 8.10 - Final Benchmark Test Run Plan
Appendix 8.11 - Final Baseline Test Run Plan
Appendix 8.12 - Pilot Testing Time Line

APPENDIX 8.1

PROCESS FLOW DIAGRAM

**(Consisting of 2 pages
Including coversheet)**


LEGEND
PRELIMINARY
AREVA Federal Services LLC
PRECONCEPTUAL
CRYSTALLIZATION SYSTEM
PROCESS FLOW DIAGRAM
C-0118-010

APPENDIX 8.2

PROCESS AND INSTRUMENTATION DIAGRAM

**(Consisting of 2 pages
Including coversheet)**

SAVANNAH RIVER NATIONAL LABORATORY

PROPRIETARY

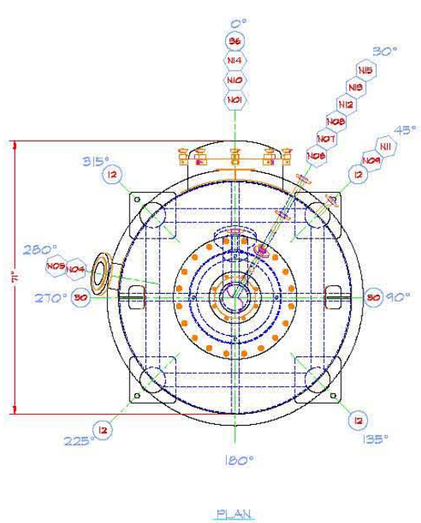
DRAWING EES-23164-M6-001

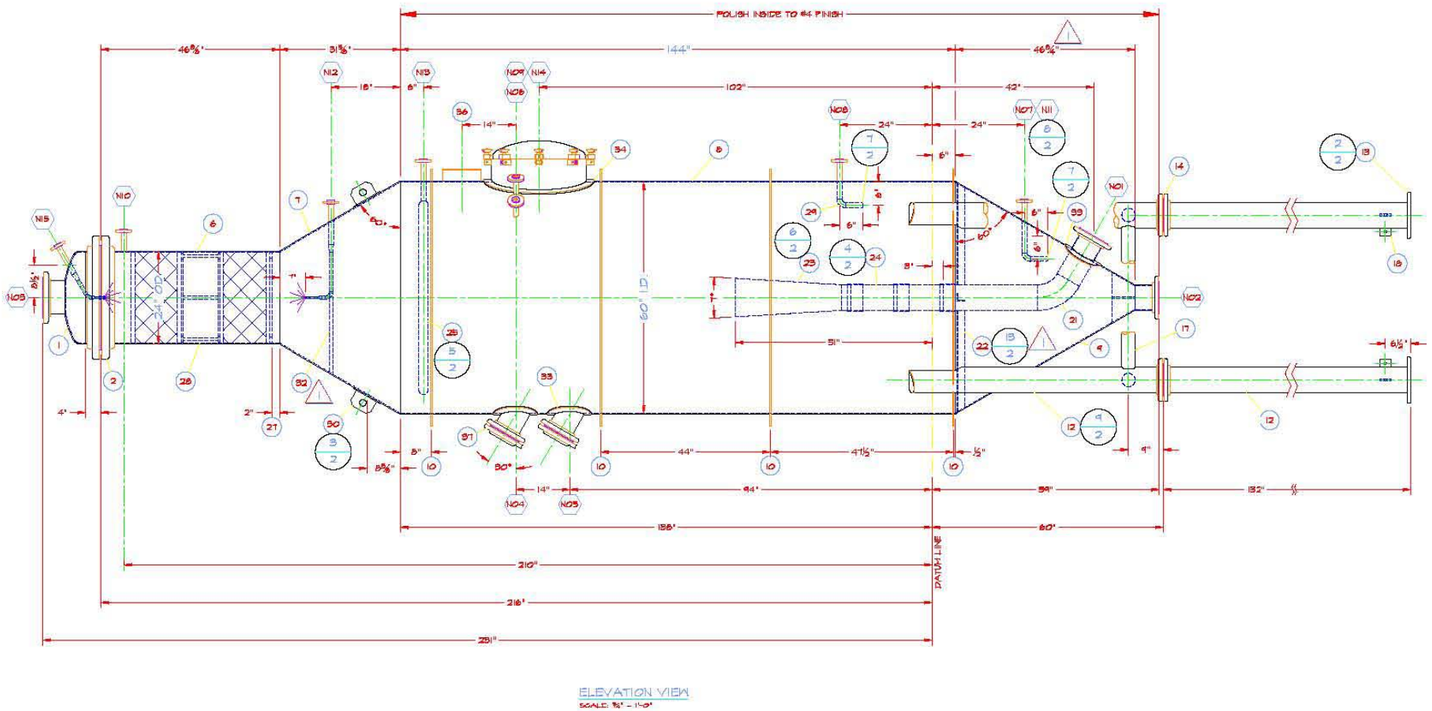
FRACTIONAL CRYSTALLIZATION PILOT SCALE FACILITY P&ID

APPENDIX 8.3

NORTHWEST COPPER CRYSTALLIZER DRAWING

**(Consisting of 2 pages
Including coversheet)**





SPECIFICATIONS

DESIGN PRESSURE	15/-15 psig
HYDRO-TEST PRESSURE	19.5 psig (at top)
DESIGN TEMPERATURE	250° F.
CODE	ASME SECTION VIII, DIVISION 1, 2004 EDITION, 2006 ADDENDA
CODE STAMP (National Board No.)	NOT REQUIRED
NON-DESTRUCTIVE TESTING	RADIOGRAPHIC TEST
RADIOGRAPH	SPOT RT PER UW-52, APPENDIX 4
JOINT EFFICIENCY	PER UW-12(b)
CORROSION ALLOWANCE	0.06"
INSPECTION OPENING	PER UG-46(a)
IMPACT TESTING	NOT REQUIRED PER UHA-51(d)(g) & UCS-66(b)
POSTWELD HEAT TREATING	NOT REQUIRED PER UCS-56 & UHA-32
MATERIAL: BOLTS/STUDS/NUTS	SA-193 B7/SA-194 2H, (ZINC ELECTROPLATED)
MATERIAL: GASKETS	1/8" 304L SS SPIRAL WOUND W/GRAPHITE FILLER
INSULATION	BY OTHERS

VESSEL

15/-15 psig
19.5 psig (at top)
250° F.
ASME SECTION VIII, DIVISION 1, 2004 EDITION, 2006 ADDENDA
NOT REQUIRED
RADIOGRAPHIC TEST
SPOT RT PER UW-52, APPENDIX 4
PER UW-12(b)
0.06"
PER UG-46(a)
NOT REQUIRED PER UHA-51(d)(g) & UCS-66(b)
NOT REQUIRED PER UCS-56 & UHA-32
SA-193 B7/SA-194 2H, (ZINC ELECTROPLATED)
1/8" 304L SS SPIRAL WOUND W/GRAPHITE FILLER
BY OTHERS

BILL OF MATERIALS

MK	THICK/SIZE	MATERIAL	HEAT NO.	DESCRIPTION	QTY
1	1/2"	SA-240 304L		70" HEAD - 24" O.D., ASME F&D w/ 1½" S.F. (0.22" min.)	1
2	STD	SA-182 304L		24" 150# ANSI RF SO FLANGE	2
3	1½"Ø	see spec.		STUDS	20
4	1¼"Ø	see spec.		NUTS	40
5	1/8"	see spec.		GASKET	1
6	1/2"	SA-240 304L		24" OD SHELL	1
7	1/2"	SA-240 304L		UPPER CONE	1
8	1/2"	SA-240 304L		60" ID SHELL	1
9	1/2"	SA-240 304L		LOWER CONE	1
10	5/8" X 5"	SA-36		STIFFENING RING	4
11					
12	6" SCH 40	SA-53B		SUPPORT LEG	4
13	5/8"	SA-36		SUPPORT LEG BASE PLATE	4
14	STD	SA-105		SUPPORT LEG FLANGE - 6" 150# ANSI RFSO	8
15					
16					
17	SCH 40	SA-53B		HORIZONTAL BRACE - 3" SCH 40 PIPE	4
18	1/2"	SA-36		LEG BOTTOM CLIP - 2"X3"	8
19					

BILL OF MATERIALS

MK	THICK/SIZE	MATERIAL	HEAT NO.	DESCRIPTION	QTY
20	5/16"	SA-240 304L		VORTEX BREAKER	4
21	SCH 10	SA-403 304L		DRAFT TUBE 60° LR ELBOW - CUT FROM 8" 90°	1
22	1/4"	SA-479 304L		DRAFT TUBE SUPPORT - 3"x3"x1/4" ANGLE	4
23	5/16"	SA-240 304L		DRAFT TUBE CONE - 10"OD X 5/8"OD	1
24	see detail	SA-240 304L		DRAFT TUBE EXTENSION ASSY	2
25	2" SCH 40	SA-312 304L		INTERNAL SPRAY RING AT N18	1
26	1/2"	SA-240 304L		INTERNAL SPRAY RING SUPPORTS	4
27	1/2" X 1/2"	SA-479 304L		MESH SUPPORT RING	8
28	3/4"Ø	SA-479 304L		MESH SPACER ROD	4
29	SCH 40	SA-403 304L		INTERNAL 1" PIPE 90° ELBOW AT N07 & N08	2
30	1/2"	SA-36		LIFTING LUGS	3
31	1/2"	SA-36		LIFTING LUG REPADS 6"X8"	2
32	1/2" X 1"	SA-479 304L		LOWER MESH SPRAY SUPPORT	1
33	1/2" X 2"	SA-240 304L		NOZZLE REPAD AT N01, 04 & N05	3
34	5/8" X 3"	SA-240 304L		NOZZLE REPAD AT N14	1
35					
36	5/16"	SA-240 316L		NAMEPLATE BRACKET (12" X 12" TEE w/ 5" LEGS)	1
37	STD	316L SS		6" PRESSURE PROD. SIGHT GLASS, MODEL L FLANGE W/SPRAY RING	2

CONNECTION SCHEDULE

MK	SIZE	RATE LB.	NECK	FLG	NECK	FLG	HEAT NO.	DESCRIPTION	TYPE
			MATERIAL		THICK				
N01	6" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		LIQUOR INLET	RPSO
N02	6" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		LIQUOR OUTLET	RPSO
N03	8" NPS	150#	SA-240 304L	SA-182 304L	1/4"	STD		VAPOR OUTLET	RPSO
N04	8" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		SIGHT GLASS	RPSO
N05	6" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		SIGHT GLASS	RPSO
N06	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		LEVEL CONNECTION	RPSO
N07	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		LEVEL CONNECTION	RPSO
N08	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		DENSITY CONNECTION	RPSO
N09	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		PRESSURE CONNECTION	RPSO
N10	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		PRESSURE DIFF	RPSO
N11	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		TEMPERATURE	RPSO
N12	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		LOWER MESH SPRAY	RPSO
N13	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		MASH RAIL	RPSO
N14	24" OD	10Ø#	SA-240 316L	----	½"	---		MANWAY, TIONA BETTS	SPEC.
N15	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		UPPER MESH SPRAY	RPSO

XXXX
CERTIFIED BY:
NORTHWEST COPPER WORKS, INC.
PORTLAND, OREGON 97227
SHELL: MAWP 15 PSIG @ 250 °F
SHELL: MAEP 15 PSIG @ 250 °F
RT-3 SHELL: MDMT -40 °F @13/-15PSIG
SERIAL NO.: 7007 YEAR BUILT: 2007
P.O. NO.: 0611002 EQUIP. NO.: later

SWENSON

Swenson Technology, Inc.
23000 Whiting Way, Monee, IL 60449-8460
Phone: 708-367-2300 ---- Fax: 708-367-2326

TYPICAL BOLTHOLE LAYOUT

NOTE:
ALL FLANGE BOLT HOLES TO STRADDLE VESSEL MAJOR CENTERLINES.

CONSTRUCTION NOTES

1. ALL NOZZLES TO PROJECT 6" FROM INSIDE VESSEL, UNLESS NOTED OTHERWISE
2. ALL WELDS TO BE POWER WIRE BRUSHED WITH SS WIRE BRUSH.
3. COVER AND PROTECT ALL NOZZLES AND OPENINGS FOR SHIPMENT WITH PLASTIC COVERS OR PLYWOOD DISCS SEALED WITH DUCT TAPE
4. MARK HEAT NUMBERS ON ALL PIECES AND INDICATE IN BILL OF MATERIAL.
5. 1/8" NPT VENT HOLE REQUIRED IN ALL NOZZLE REINFORCEMENT PLATES. AIR TEST ALL NOZZLE REINFORCEMENT PADS.
6. DO NOT INTERSECT OR COVER ANY SHELL SEAMS WITH NOZZLES, DOUBLERS, ETC.
7. PAINT ALL C-STL WITH ONE COAT OF RED-OXIDE PRIMER.
8. INTERNAL SURFACE TO BE POLISHED TO #4 FINISH WHERE SHOWN.

REVISIONS

NO	DESCRIPTION	BY	APD BY	DATE
1	GENERAL REVISIONS	SHT	MJV	08/08/07

ESTIMATED WEIGHT: (IN LBS)
OPERATING - 21,490 lbs.
FULL WATER - 23,100 lbs.
EMPTY - 5,600 lbs.

SWENSON DWG. #SD-0611102-1

NORTHWEST COPPER WORKS, INC.
1808 N. RIVER STREET * PORTLAND, OREGON * 97227

60" ID X 144" LONG CRYSTALLIZER VESSEL

SWENSON TECHNOLOGY
23000 WHITING WAY
MONEE, IL 60449

EQUIP. NO. 7
P.O. NO. 0611002
SCALE AS NOTED
JOB NO. 7007
DATE 08/04/07
CHKD. BY MJV
DWN. BY SHT
APPD. BY MJV
DWG. NO. D-7007
REV 1
SHT. NO. 1 OF 2

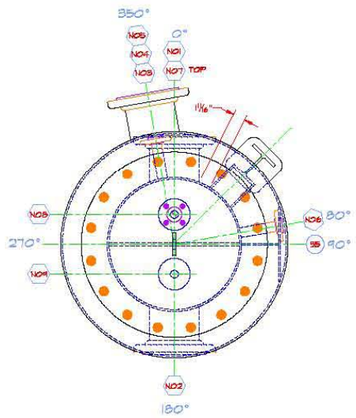
FRACTIONAL CRYSTALLIZATION PROJECT SAVANNAH RIVER, SA

REF. DWG SD-0611101-00

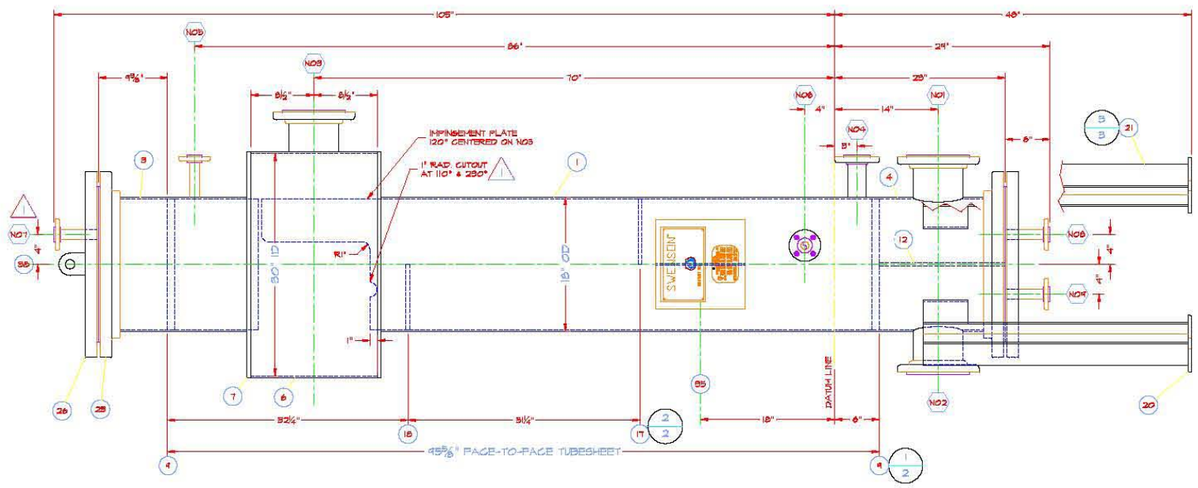
APPENDIX 8.4

NORTHWEST COPPER REBOILER DRAWING

(Consisting of 2 pages
Including coversheet)



PLAN



ELEVATION VIEW
SCALE: ½" = 1'-0"

SPECIFICATIONS

	SHELLSIDE	TUBESIDE
DESIGN PRESSURE	15/-15 psig	15/-15 psig
HYDRO-TEST PRESSURE	19.5 psig (AT TOP)	19.5 psig (AT TOP)
DESIGN TEMPERATURE	250° F.	140° F.
CODE	ASME SECTION VIII, DIVISION 1, 2004 EDITION, 2006 ADDENDA	
CODE STAMP (National Board No.)	NOT REQUIRED	NOT REQUIRED
NON-DESTRUCTIVE TESTING	RADIOGRAPHIC TEST	RADIOGRAPHIC TEST
RADIOGRAPH	SPOT RT PER UW-52 & APP. 4	SPOT RT PER UW-52 & APP. 4
JOINT EFFICIENCY	PER UH-12(b)	PER UH-12(b)
CORROSION ALLOWANCE	0.04"	0.04"
INSPECTION OPENING	NOT REQUIRED PER UG-46(a)	REQUIRED PER UG-46(f)
IMPACT TESTING	NOT REQUIRED PER UHA-51(d)(1)(g) & UCS-66	
POSTWELD HEAT TREATING	NOT REQUIRED PER UHA-32	
MATERIAL, BOLTS/STUDS/NUTS	SA-193 B7/SA-194 2H, (ZINC ELECTROPLATED)	
MATERIAL, GASKETS	⅛" SCHL SPIRAL WOUND 316/GRAPHITE FILLER	
INSULATION	BY OTHERS	

BILL OF MATERIALS

MK	THICK/SIZE	MATERIAL	HEAT NO.	DESCRIPTION	QTY
1	½"	SA-240 304L		SHELL	A/R
2					
3	½"	SA-240 304L		RETURN CHANNEL SECTION	1
4	½"	SA-240 304L		INLET/OUTLET CHANNEL SECTION	1
5					
6	½"	SA-240 304L		PLENUM SHELL	1
7	½"	SA-240 304L		PLENUM CLOSURES	2
8	⅜"	SA-240 304L		BACKING STRIP	2
9	1"	SA-240 304L		TUBESHEETS	2
10	1½"Ø	SA-213 TP304L		TUBES-1½"Ø X 16 BWG X 8'-0" LONG	40
11	⅜"Ø	SA-479 304L		TIERODS	8
12	½"	SA-240 304L		PASS PARTITION BAFFLE	1
13	1" x ½"	SA-240 304L		PASS PARTITION PACKING RETAINING BARS	2
14	⅜" SQ.	EXPANDED PTFE		PASS PARTITION PACKING	1
15					
16				DELETED	
17	½"	SA-240 304L		TUBE SUPPORT BAFFLE "A"	1
18	½"	SA-240 304L		TUBE SUPPORT BAFFLE "B"	1
19					

BILL OF MATERIALS

MK	THICK/SIZE	MATERIAL	HEAT NO.	DESCRIPTION	QTY
20	½"	SA-36		LEG BASEPLATE	4
21	STD	SA-36		AXIS SUPPORT LEG	4
22	½"	SA-240 304L		LEG MOUNT GUSSET	8
23					
24					
25	STD	SA-182 304L		CHANNEL FLANGES - 18" ANSI 150# RFSO	2
26	STD	SA-182 304L		CHANNEL BLIND FLANGES - 18" ANSI 150#	2
27	1⅛"Ø	888 spec		STUDS - CHANNEL BODY FLANGE	52
28	1⅛"Ø	888 spec		NUTS - CHANNEL BODY FLANGE	84
29	1⅛" Thk.	888 spec		CHANNEL FLANGE GASKET	2
30					
31					
32					
33					
34					
35	⅜"	SA-240 304L		NAMEPLATE BRACKET (12" x 12" TEE w/ 3" LEG)	1
36					
37					
38	⅜"	SA-240 304		TOP COVER LIFTING LUG	1

CONNECTION SCHEDULE

MK	SIZE	RATE LB.	NECK	FLG	THICK NECK	THICK FLG	HEAT NO.	DESCRIPTION	TYPE
NO1	6" NPS	150#	SA-312 304L	SA-182 304L	Sch10S	STD		LIQUOR INLET	RFSO
NO2	6" NPS	150#	SA-312 304L	SA-182 304L	Sch10S	STD		LIQUOR OUTLET	RFSO
NO3	6" NPS	150#	SA-312 304L	SA-182 304L	Sch10S	STD		STEAM INLET	RFSO
NO4	2" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		CONDENSATE OUTLET	RFSO
NO5	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		UPPER SHELL VENT	RFSO
NO6	1" NPS	150#	SA-312 304L	SA-182 304L	Sch40	STD		LOWER SHELL VENT	RFSO
NO7	1" NPS	150#	SA-312 304L	SA-182 304L	Sch80S	STD		TOP VENT	RFSO
NO8	1" NPS	150#	SA-312 304L	SA-182 304L	Sch80S	STD		DRAIN	RFSO
NO9	1" NPS	150#	SA-312 304L	SA-182 304L	Sch80S	STD		DRAIN	RFSO

XXXX
CERTIFIED BY:
NORTHWEST COPPER WORKS, INC.
PORTLAND, OREGON 97227

RT-3

SHELL:	MAWP	15 PSIG @ 250°F
SHELL:	MAEP	15 PSIG @ 250°F
SHELL:	MDMT	48°F @15/-15PSIG
TUBE:	MAWP	15 PSIG @ 140°F
TUBE:	MAEP	15 PSIG @ 140°F
TUBE:	MDMT	48°F @15/-15PSIG

SERIAL NO.: 7008 YEAR BUILT: 2007
P.O. NO.: 0617002 EQUIP. NO.: 1&1A

SWENSON

Swenson Technology, Inc.
2000 Phiting Way, Monee, IL 60449-8349
Phone: 708-587-3300 ~~~ Fax: 708-587-3333

TYPICAL BOLTHOLE LAYOUT

NOTE:
ALL FLANGE BOLT HOLES TO STRADDLE VESSEL MAJOR CENTERLINES.

CONSTRUCTION NOTES

1. ALL NOZZLES TO PROJECT 6" FROM INSIDE VESSEL, UNLESS NOTED OTHERWISE
2. ALL WELDS TO BE POWER WIRE BRUSHED WITH SS WIRE BRUSH.
3. COVER AND PROTECT ALL NOZZLES AND OPENINGS FOR SHIPMENT WITH PLASTIC COVERS OR PLYWOOD DISCS SEALED WITH DUCT TAPE
4. MARK HEAT NUMBERS ON ALL PIECES AND INDICATE IN BILL OF MATERIAL.
5. 1/8" NPT VENT HOLE REQUIRED IN ALL NOZZLE REINFORCEMENT PLATES. AIR TEST ALL NOZZLE REINFORCEMENT PADS.
6. DO NOT INTERSECT OR COVER ANY SHELL SEAMS WITH NOZZLES, DOUBLERS, ETC.

REVISIONS

NO	DESCRIPTION	SHT REV BY	MJV APD BY	09/05/07 DATE
1	GENERAL REVISIONS	SHT	MJV	09/05/07

SWENSON DWG. #SD-0617103-1

ESTIMATED WEIGHT: (IN LBS)

OPERATING =	2,650 lbs.
FULL WATER =	3,200 lbs.
EMPTY =	2,175 lbs.

NORTHWEST COPPER WORKS, INC.
1808 N. RIVER STREET * PORTLAND, OREGON * 97227

18" OD X 7'-11⅝" TS-TS
FRACTIONAL REBOILER

SWENSON TECHNOLOGY 28000 PHITING WAY MONEE, IL 60449		EQUIP. NO.	7
		P.O. NO.	0617002
SCALE	AS NOTED	JOB NO.	7008
DATE	08/04/07	DWG. NO.	D-7008
DRN. BY	SHT	CHKD. BY	MJV
		APPD. BY	MJV
		REV	1
		SHT. NO.	1 OF 3

FRACTIONAL CRYSTALLIZATION PROJECT REF. DWG SD-0617103-00
SAVANNAH RIVER, GA

APPENDIX 8.5

PILOT INSTRUMENT LIST

**(Consisting of 3 pages
Including coversheet)**

Instrumentation List for Fractional Crystallization Test Facility

Function	Loop ID	TR#	Quantity Measured	Post Test Calibration	Reliable During Testing	Measuring Device	Local /DAS	Range	Tolerance	Vendor, Model No, and Other Information	Need Spare	Calibration Status	Date Walkdown	Date Oper. Verification	Cost	Cost Avoided	PO	Resp*	Date Ordered	Delivery Date	Status
	CO		**Condensate System**																		
TE	C001	TR-03858	Condensate Tank Temp.			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
CT	C002	TR-03884	Condensate Conductivity Transmitter				DAS	0-1000 uS/cm	2%rdg+6uS/cm	Omega CDCN-91AC		Calibrated						TS	on hand		on hand
CE	C002	TR-03884	Condensate Conductivity Probe				DAS	0-1000 uS/cm	2%rdg+6uS/cm	CDCE-90-01 Cell		Calibrated						TS	on hand		on hand
LAH	C003	N/A	Condensate Tank High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC			received
FI	C004	TR-00122	Condensate Flow Rate to Crystallizer Spray Ring			Rotameter	Local	0-5 gpm	+/- 0.35 gpm			Calibrated						SS	on hand		on hand
FI	C005		Condensate Flow to Crystallizer Lower Demister			Rotameter	Local	0-20 gph	+/- 0.4 gph	Fischer & Porter 10A6132M								SS	on hand		on hand
FI	C006	TR-20269	Condensate Flow to Crystallizer Upper Demister			Rotameter	Local	0-2 gpm	+/- 0.15 gpm			Calibrated						SS	on hand		on hand
FI	C007	TR-20268	Condensate Flow for Desuperheater			Rotameter	Local	0-2 gpm	+/- 0.15 gpm			Calibrated						SS	on hand		on hand
LC	C008	N/A	Reboiler Condensate receiver Level Control							Dwyer, PCP-10		Not Needed			$141			JG	06/18/07		received
LI	C008	N/A	Reboiler Condensate receiver sight glass							McMaster-Carr 3721K78		Not Needed			$56		2P5943-1	JG	06/18/07		received
LSH	C008	N/A	Reboiler Condensate receiver level			Float Switch	Local			Dwyer/WE Anderson, F7-HPS-21		N/A			$29			JG	06/18/07		received
LSL	C008	N/A	Reboiler Condensate receiver level			Float Switch	Local			Dwyer/WE Anderson, F7-HPS-21		N/A			$29			JG	06/18/07		received
FI	C009	TR-03887	Centrifuge Rind Wash Water Totalizer				Local	0.22-22 gpm	2.5% of rdg	Omega FTB80007A		Calibrated			$163			TS	08/01/07		received
TE	C010	TR-03908	Temp.of Condensate Feed to Product Disolver			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
FT	C011	TR-03563	Condensate to Product Dissolver Flow Rate			Magnetic FM	DAS	0-2 gpm	4% of reading		Y	Calibrated				$2,500		JC	on hand		on hand
PT	C012	TR-03717	Interstage Jet Pump Pressure			Press Transd	DAS	0-15 psia	0.5% +/- FS	`		Calibrated						JC	on hand		on hand
FI	C013	TR-03510	Condensate Purge Flow to Crystallizer Nozzle #10			Rotameter	Local	0-2 gph	4% of reading			Not Needed						SS	on hand		on hand
FI	C014	TR-03658	Condensate Purge Flow to Crystallizer Nozzle #6			Rotameter	Local	0-2 gph	4% of reading			Not Needed						SS	on hand		on hand
FI	C015	TR-03878	Condensate Purge Flow to Crystallizer Nozzle #7			Rotameter	Local	0-2 gph	4% of reading			Not Needed						SS	on hand		on hand
FI	C016	TR-03879	Condensate Purge Flow to Crystallizer Nozzle #8			Rotameter	Local	0-2 gph	4% of reading			Not Needed						SS	on hand		on hand
PI	C017	N/A	Condensate Header Pressure			Pressure Gage	Local	0-100 psig		Moore Products Co. #3994		Not Needed						JC	06/29/07		on hand
FT	C018	TR-03677	Condensate Flow to HLW Receipt Tank			Magnetic FM	DAS	0-2 gpm	4% of reading		Y	Calibrated				$2,500		JC	on hand		on hand
LC	C019	N/A	Condensate Tank Level Control							Omega LV611-P		Not Needed			$33			TS	07/30/07		received
FI	C020	TR-03880	Condensate Purge Flow to PD Density Transmitter			Rotameter	Local	0-2 gph	4% of reading	Brooks Sho-Rate		Not Needed						SS	on hand		on hand
FI	C021	TR-03881	Condensate Purge Flow to PD Density Transmitter			Rotameter	Local	0-2 gph	4% of reading	Brooks Sho-Rate		Not Needed						SS	on hand		on hand
PI	C022	N/A	Bottom Spray Nozzle supply pressure			Pressure Gage	Local	Vac-60 psig	.2% of reading	McMaster Carr #3854SK16		Not Needed						JC			
PI	C023	N/A	Top Spray Nozzle supply pressure			Pressure Gage	Local	Vac-60 psig	.2% of reading	McMaster Carr #3854SK16		Not Needed						JC			
FI	C024	TR-20271	Condensate Flow Rate to Crystallizer Lower Nozzle			Rotameter	Local	0-2 gpm	+/- 0.15 gpm			Calibrated						SS	on hand		on hand
	CW		**Cooling Water System**																		
TE	CW01	TR-03857	Condenser Cooling Water Inlet Temp.			Thermocouple	DAS	32-212 F	3.6 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
FT	CW02	TR-03687	Condenser Cooling Water Flow Rate			Magnetic FM	DAS	0-200 gpm	4% of reading			Calibrated						SS	on hand		on hand
TE	CW03	TR-03846	Condenser Cooling Water Outlet Temp.			Thermocouple	DAS	32-212 F	3.6 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
FI	CW05	N/A	Jet Pump Cooling Water 1st Stage Flow Rate			Rotameter	Local	0-20 gpm	4% of reading			Not Needed			$156			SS	10/16/07	11/30/07	received
TE	CW06	TR-03899	Jet Pump 1st. Stage Cooling Water Temp. outlet			Thermocouple	DAS	32-212 F	3.6 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
FI	CW07	N/A	Jet Pump Cooling Water 2nd Stage Flow Rate			Rotameter	Local	0-20 gpm	4% of reading			Not Needed			$156			SS	10/16/07	11/30/07	received
TE	CW08	TR-03901	Jet Pump 2nd. Stage Cooling Water Temp. outlet			Thermocouple	DAS	32-212 F	3.6 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
PI	CW09	N/A	Seal water header pressure			Pressure Gage	Local	0-100 psig		Glycerin-Filled McMaster Carr #4088K511		Not Needed			$85			JC	06/29/07	07/09/07	received
TE	CW10	TR-03856	Seal Water Temperature			Thermocouple	DAS	32-212 F	3.6 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
FI	CW11	N/A	Circulation pump seal water flow			Rotameter	Local	0-5 gpm	4% of reading	Brooks 1305FJ1BCJ1AA		Not Needed						MR			on hand
PI	CW12	N/A	Circulation Pump seal water pressure			Pressure Gage	Local	0-100 psig		Glycerin-Filled McMaster Carr #4088K511											
FI	CW13	N/A	Drawoff pump seal water flow			Rotameter	Local	0-5 gpm	4% of reading	Dwyer RSF122		Not Needed						MR			on hand
PI	CW14	N/A	Drawoff pump seal water pressure			Pressure Gage	Local	0-100 psig		Glycerin-Filled McMaster Carr #4088K511		Not Needed			$85			JC	06/29/07	07/09/07	received
	FR		**Feed/Receipt System**																		
FT	FR01	TR-03678	Feed Flow Rate			Magnetic FM	DAS	0-2 gpm	4% of reading		Y	Calibrated				$2,500		JC	on hand		on hand
PI	FR02	N/A	Feed Pressure			Pressure Gage	Local	0-30 psig		Glycerin-Filled McMaster Carr #4088K511		Not Needed			$85			JC	06/29/07	07/09/07	received
TE	FR03	TR-03851	Feed Temperature			Thermocouple	DAS	32-212 F	3.6 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
LAH	FR04A	N/A	Feed Tank A High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
LAH	FR04B	N/A	Feed/LAW Tank B High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
LAH	FR04C	N/A	Feed/LAW Tank C High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
LAH	FR04D	N/A	Feed/LAW Tank D High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
LAH	FR04E	N/A	HLW Tank E High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
SC	FR05	N/A	Feed/Receipt Pump 1 VFD			VFD	Local					Not Needed						JC	on hand		on hand
LI	N/A	N/A	Feed Tank A volume			Tank Markings	Local			Mark side of tank at 100 gallon intervals		Calibrated							N/A		
LI	N/A	N/A	Feed Tank B volume			Tank Markings	Local			200 gal major, 20 gal minor intervals		Calibrated							N/A		
LI	N/A	N/A	Feed Tank C volume			Tank Markings	Local			Mark side of tank at 100 gallon intervals		Calibrated							N/A		
LI	N/A	N/A	Feed Tank D volume			Tank Markings	Local			Mark side of tank at 100 gallon intervals		Calibrated							N/A		
LI	N/A	N/A	Feed Tank E volume			Tank Markings	Local			Mark side of tank at 100 gallon intervals		Calibrated							N/A		
	PD		**Product Dissolver System**																		
TE	PD01	TR-03862	Product Dissolver Tank Temp.			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12	Y	Calibrated			$26			JC	07/12/07	07/24/07	received
TC	PD02	N/A	Product Disolver Tank Heater Controller							Omega CN7823 / SSR3PH660DC30	Y	Not Needed			$244			DF	07/25/07		received
TE	PD02	TR-03847	Product Dissolver Tank Temp. for Heater Controller			Thermocouple	Local	32-212 F	1.8 +/- F	Omega EQ316SS-116(G)-12 1/16" E Type T/C		Calibrated			$26			TS	08/01/07		received
TE	PD03	TR-03865	Product Dissolver Tank Temp. for Heater Shutdown			Thermocouple	Local	32-212 F	3.6 +/- F	Omega EQ316SS-116(G)-12 1/16" E Type T/C		Calibrated			$26			TS	08/01/07		received
TSC	PD03	N/A	Product Disolver Tank Heater Safety Shutdown							Omega CN3271-R1		Test			$200			DF	07/25/07		received
PI	PD04	N/A	Product Dissolver Filter Inlet Pressure			Pressure Gage	Local	0-60 psig		Glycerin-Filled McMaster Carr #3813K7		Not Needed			$85			JC	06/29/07	07/09/07	received
TE	PD05	TR-03853	Product Dissolver Centrifuge Feed Temperature			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
DT	PD06	TR-03490	Product Dissolver Density (Sp.Gr.)			Diff. Press Trans	DAS	0-100 in wc		Rosemount		Calibrated						ZQ	on hand		on hand
LC	PD07	N/A	Product Dissolver Tank Level Controller				DAS			Omega LVCN-51		Verify			$425			TS	07/30/07		received
LE	PD07	TR-03919	Product Dissolver Tank Level			Ultrasonic	DAS	0.3 - 6 ft	0.125 in	Omega LVU91		Calibrated			$495			TS	07/30/07		received
LAH	PD08	N/A	Product Dissolver Tank High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
FT	PD09	TR-03276	Product Dissolver Tank Purge Flow			Magnetic FM	DAS	0-2 gpm	4% of reading			Calibrated				$2,500		JC	on hand		on hand
FT	PD10	TR-03704	Product Dissolver Centrifuge Feed Flow Rate			Magnetic FM	DAS	0-5 gpm	4% of reading			Calibrated				$2,500		JC	on hand		on hand

PI	PD11	N/A	Product Dissolver Filtrate Pressure			Pressure Gage	Local	0-30 psig		Glycerin-Filled McMaster Carr #3813K7		Not Needed			$85			JC	06/29/07	07/09/07	received
HS	PD12	N/A	PD Filter Backwash Switch			N/A						N/A						TS			on hand
	RW		**Recycle Wash System**																		
TE	RW01	TR-03906	Recycle Wash Tank Temp.			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
TC	RW02	N/A	Recycle Wash Tank Heater Controller							Omega CN7823 / SSR3PH660DC30		Not Needed			$244			DF	07/25/07		received
TE	RW02	TR-03842	Recycle Wash Tank Temp. for Heater Control			Thermocouple	Local	32-212 F	1.8 +/- F	Omega EQ316SS-116(G)-12 1/16" E Type T/C		Calibrated			$26			TS	08/01/07		received
TE	RW03	TR-03866	Recycle Wash Tank Temp. for Heater Shutdown			Thermocouple	Local	32-212 F	3.6 +/- F	Omega EQ316SS-116(G)-12 1/16" E Type T/C		Calibrated			$26			TS	08/01/07		received
TSC	RW03	N/A	Recycle Wash Tank Heater Safety Shutdown							Omega CN3271-R1		Test			$200			DF	07/25/07		received
PI	RW04	N/A	Recycle Wash Pressure			Pressure Gage	Local	0-60 psig		Glycerin-Filled McMaster Carr #3813K7		Not Needed			$85			JC	06/29/07	07/09/07	received
TE	RW05	TR-03843	Recycle Wash Centrifuge Feed Temperature			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
LAH	RW06	N/A	Recycle Wash Tank High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC			received
LE	RW07	TR-03918	Recycle Wash Tank Level			Ultrasonic	DAS	0.3 - 6 ft	0.125 in	Omega LVU91		Calibrated			$495			TS	07/30/07		received
	SL		**Slurry System**																		
IT	SL01	N/A	Circulation Pump Current				DAS	0-20 amps				Vendor						Areva		10/03/07	received
SC	SL01	N/A	Circulation Pump VFD				DAS					N/A						Areva		10/03/07	received
ST	SL01	N/A	Circulation Pump Speed				DAS	0-3550 rpm				Verify						Areva		10/03/07	received
TDW	SL02	TR-03889	Slurry Temp. rise across Reboiler			Diff. T/couple	DAS	0-20 C	+/- 0.25 C	Omega 5TC-GG-E-20-72	Y	Calibrated			$49			TS			received
FT	SL03	TR-03892	Circulation Flow Rate			Ultrasonic FM	DAS	0-1000 gpm	2 %rdg+0.2 fps	GE TransPort PT878		Calibrated						ZQ	07/27/07		received
TW	SL04	TR-03861	Crystallizer Vessel Temperature			Thermocouple	DAS	32-212 F	1.8 +/- F	1/4", 12"L, Type E, Omega # GEQSS-14(G)-12	Y	Calibrated			$30			JC	07/12/07	07/24/07	received
DT	SL05	TR-03493	Crystallizer Slurry Density			Diff. Press Trans	DAS	0-100 in wc	0.5% +/- FS	Rosemount		Calibrated						ZQ	on hand		on hand
LT	SL06	TR-03691	Crystallizer Vessel Level			Diff. Press Trans	DAS	0-200 in wc	0.5% +/- FS	Rosemount		Calibrated						ZQ	on hand		on hand
PT	SL07	TR-03885	Crystallizer Vessel Pressure			Pressure Trans	DAS	0-15 psia	0.5% +/- FS	Omega, PX4200-30VACI		Calibrated						ZQ	08/09/07		received
PDT	SL08	TR-03106	Pressure Drop across Filter Pads			Diff. Press Trans	DAS	0-30 in wc	0.5% +/- FS	Rosemount		Calibrated						ZQ	on hand		on hand
CE	SL10	TR-03891	Slurry Conductivity in Drawoff Line			Cond Probe	DAS	0-2 S/cm		Omega CDE-45T1 electrodless cond. sensor		Calibrated			$405			JS	08/27/07		received
CT	SL10	TR-03891	Slurry Conductivity in Drawoff Line			Transmitter	DAS			Omega CDTX-45T1 Conductivity analyzer		Calibrated			$675			JS	08/27/07		received
PI	SL11	N/A	Draw Off Line Pressure			Pressure Gage	Local	vac-30 psig	1.5% FS	Noshok 25-400 30"Hg/30 psig + McMaster isolator 4334K31		Not Needed						ZQ	on hand		on hand
TE	SL12	TR-03845	Slurry Temp. to Centrifuge			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
FT	SL14	TR-03601	Draw Off Return Flow Rate			Magnetic FM	DAS	0-100 gpm	4% of reading	TR-03601		Calibrated				$2,500		JC	on hand		on hand
FT	SL15	CDS-4714	Draw Off Loop Flow Rate (Ultrasonic)			Ultrasonic FM	DAS	0-60 gpm	1.5%rdg+0.15fps	Controlotron System 990		Calibrated						MF			
FI	SL16	N/A	Air bleed to the steam jet vacuum pump			Rotameter	Local	0-2 scfm		Fischer-Porter 10A6131M		Not Needed						TS	on hand		
SC	SL17	N/A	Draw off pump VFD																		
	ST		**Steam System**																		
FE	ST01	N/A	Reboiler Steam Flow Orifice			Orifice				Lamdasquare, Oripac 5300 (1.1" ID)		ID Verified			$288			JG	06/19/07	06/27/07	received
FT	ST01	TR-03688	Reboiler Steam Flow Rate			DP across orifice	DAS	0-30 in wc	0.5% +/- FS	Rosemount		Calibrated						ZQ	on hand		on hand
TE	ST02	TR-03904	Steam Header Temperature			Thermocouple	DAS	32-392 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
PI	ST03	TR-03886	Steam Header Pressure			Pressure Gage	Local	0-160 psig		Glycerin-Filled McMaster Carr #4088K511		Calibrated			$85			JC	06/29/07	07/09/07	received
TW	ST04	TR-03905	Steam Temperature after Desuperheater			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
TW	ST05	TR-03851	Condensate Temp. at Reboiler exit			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
PIC	ST06	N/A	Reboiler Steam Process Controller							Schubert & Salzer, 2504 Process Controller		Not Needed			$501			JG	06/28/07	08/10/07	received
PE	ST06	TR-03883	Reboiler Shell Steam Pressure Element			Pressure Transd	DAS	0-15 psia	0.5% +/- FS	Omega, PX4200-30VACI		Calibrated			$303			JG	06/18/07	06/20/07	received
PT	ST06	TR-03883	Reboiler Pressure Transmitter							Omega, DP25B-E		Calibrated			$325			JG	06/18/07	06/20/07	received
ZC	ST06	N/A	Reboiler Steam Valve Position Controller					0-100% Open		Schubert and Salzer Model 8049 Positioner		Not Needed									
PI	ST07	N/A	Jet Pump Steam Pressure for 2nd. Stage			Pressure Gage	Local	0-160 psig		Glycerin-Filled McMaster Carr #4088K511		Not Needed			$85			JC	06/29/07	07/09/07	received
PI	ST08	N/A	Jet Pump Steam Pressure for 1st. Stage			Pressure Gage	Local	0-160 psig		Glycerin-Filled McMaster Carr #4088K511		Not Needed			$85			JC	06/29/07	07/09/07	received
PI	ST09	N/A	Steam Header Pressure			Pressure Gage	Local	0-160 psig		Glycerin-Filled McMaster Carr #4088K511		Not Needed									
	SW		**Spent Wash System**																		
TE	SW01	TR-03852	Spent Wash Tank Temp.			Thermocouple	DAS	32-212 F	1.8 +/- F	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12		Calibrated			$26			JC	07/12/07	07/24/07	received
TC	SW02	N/A	Spent Wash Tank Heater Control							Omega CN7823 / SSR3PH660DC30		Not Needed			$244			DF	07/25/07		received
TE	SW02	TR-03873	Spent Wash Tank Temp. for Heater Control			Thermocouple	Local	32-212 F	1.8 +/- F	Omega EQ316SS-116(G)-12 1/16" E Type T/C		Calibrated			$26			TS	08/01/07		received
TE	SW03	TR-03867	Spent Wash Tank Temp. for Heater Shutdown			Thermocouple	Local	32-212 F	3.6 +/- F	Omega EQ316SS-116(G)-12 1/16" E Type T/C		Calibrated			$26			TS	08/01/07		received
TSC	SW03	N/A	Spent Wash Tank Heater Safety Shutdown							Omega CN3271-R1		Test			$200			DF	07/25/07		received
PI	SW04	N/A	Spent Wash Pressure			Pressure Gage	Local	0-60 psig		Glycerin-Filled McMaster Carr #3813K7		Not Needed			$85			JC	06/29/07	07/09/07	received
HS	SW05	N/A	Sample Switch			Switch	Local	N/A	N/A			N/A						TS			on hand
FT	SW06	TR-03721	Spent Wash to HLW Tank Flow Rate			Magnetic FM	DAS	0-2 gpm	4% of reading	TR-03721		Calibrated				$2,500		JC	on hand		on hand
LC	SW07	N/A	Spent Wash Tank Level Controller				DAS			Omega LVCN-51		Verify			$425			TS	07/30/07		received
LE	SW07	TR-03917	Spent Wash Tank Level			Ultrasonic	DAS	0.3 - 6 ft	0.125 in	Omega LVU91		Calibrated			$495			TS	07/30/07		received
LAH	SW08	N/A	Spent Wash Tank High Level Alarm			Float Switch	Local			McMaster Carr #46515K41 Polypropylene		N/A			$15			JC	on hand		received
FT	SW09	TR-03705	Spent Wash Flow to Crystallizer			Magnetic FM	DAS	0-2 gpm	4% of reading	TR-03705		Calibrated				$2,500		JC	on hand		on hand
	* Organization or initials of responsible person in SRNL. Note that the initials will be updated as activity changes, e.g. design, specifications, procurement, inspection etc.																				
	* **Initials**: DF-Don Fisher, JC-Jerry Corbett, JG-John Gordon, JS-John Steimke, MR-Mike Restivo, SS-Susan Shouse, TS-Tim Steeper, ZQ-Zafar Qureshi																				
	Note: Red text identifies changes from the last approved P&ID drawing revision.																				
										SPARE M&TE										M&TE#	
										1/4", 12"L, Type E, Omega # GEQSS-14(G)-12										TR-03860	
										Type E, Omega # GEQSS-18(G)-12										TR-03863	
										Type E, Omega # GEQSS-18(G)-12										TR-03864	

APPENDIX 8.6

PILOT VALVE LIST

**(Consisting of 5 pages
Including coversheet)**

	Valve List for Fractional Crystallization Test Facility														
Line #	Sys	Valve No.	Type	Size	Material	Vendor, Model No., and Other Information	P&ID Loc	Date Walkdown	Date Oper. Verification	Comments	Date Order Placed	Delivery Date	Status	Cost	Cost Avoided
1	CO	1	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	V17				7/10/2007	7/17/2007	received	$20	
2	CO	2	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	W17				7/10/2007	7/17/2007	received	$7	
3	CO	3	T	1/4" Tube	S/S	Swagelok SS-1RS4	V29				6/18/2007	6/27/2007	on hand	$49	
4	CO	4	T	3/8" Tube	S/S	Swagelok SS-1VS6	V29				6/18/2007	6/27/2007	on hand	$68	
5	CO	5	S	3/8" Pipe	BRASS	AUTOMATIC SWITCH CO. #EF8210G34	V29						on hand		
6	CO	6	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	W31				7/10/2007	7/17/2007		$7	
7	CO	7	T	1/2" Pipe	SS	Parker 494F 316	F38						on hand	$200	
8	CO	8	A/A 3W	1/2" Pipe	S/S	Assured Automation Mod. C33DAX08S1E3A	F37				7/23/2007		received	$417	
9	CO	9	B	1/4" Tube	S/S	Swagelok SS-43S4	N17						on hand		$62
10	CO	10	B	3/8" Tube	S/S	Swagelok SS-43S6	Z5						on hand		$64
11	CO	11	T	3/4 Pipe	S/S	McMaster Carr #4742K14 3/4" SS Globe	Y6				7/17/2007	2/23/2007	received	$81	
12	CO	12	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AC1				7/10/2007	7/17/2007		$7	
13	CO	13	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AC5				7/10/2007	7/17/2007		$7	
14	CO	14	T	1/2" Pipe	SS	Parker 494F 316	J13						on hand	$200	
15	CO	15	T	1/4" Tube	S/S	Swagelok SS-1RS4	J14				6/15/2007	6/27/2007	received	$49	
16	CO	16	T	1/4" Tube	S/S	Swagelok SS-1RS4	J14				6/15/2007	6/27/2007	received	$49	
17	CO	17	T	1/4" Tube	S/S	Part of Brooks rotameter	M3				6/15/2007	6/27/2007	received	$49	
18	CO	18	T	1/4" Tube	S/S	Part of Brooks rotameter	M4				6/15/2007	6/27/2007	received	$49	
19	CO	19	T	1/4" Tube	S/S	Part of Brooks rotameter	M4				6/15/2007	6/27/2007	received	$49	
20	CO	20	T	1/4" Tube	S/S	Part of Brooks rotameter	M5				6/15/2007	6/27/2007	received	$49	
21	CO	21A	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	V28				7/10/2007	7/17/2007	received	$11	
22	CO	21B	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	U28				7/10/2007	7/17/2007	received	$11	
23	CO	22	T	1/4" Pipe	SS	Integral part of rotameter	U11						on hand		
24	CO	23F	B	1/2" Tube	CPVC	Whitey SS-45F8	Y27				7/10/2007	7/17/2007	received	$7	
25	CO	23G	B	1/2" Tube	CPVC	Whitey SS-45F8	Y27				7/10/2007	7/17/2007	received	$7	
26	CO	24	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AC23						received		
27	CO	25	B	1/4" Tube	S/S	Swagelok SS-43S4	S37						on hand		$62
28	CO	26	B	1/4" Tube	S/S	Swagelok SS-43S4	S38						on hand		$62
29	CO	27	T	1/4" Tube	S/S	Part mof Brooks rotameter	U38				6/15/2007	6/27/2007	received	$49	
30	CO	28	T	1/4" Tube	S/S	Part mof Brooks rotameter	U38				6/15/2007	6/27/2007	received	$49	
31	CO	29	B	1/4" Tube	S/S	Swagelok SS-43S4	T38						on hand		$62
32	CO	30A	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	Z5				7/10/2007	7/17/2007	received	$11	
33	CO	30B	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	Z6				7/10/2007	7/17/2007	received	$11	
34	CO	31	T	3/8" Tube	S/S	Swagelok SS-1VS6	U18				6/18/2007	6/27/2007	received	$68	
35	CO	32	AT	1/2" Tube	S/S	Omega PV14-B	Q29						received	$1,080	
36	CO	33	A/A 3W	1/2" Tube	S/S	Assured Automation Mod. C33DAX08S1E3A	R30				7/23/2007		received	$417	
37	CO	34	B	1/4" Tube	S/S	Swagelok SS-43S4	R31						on hand		$62
38	CO	35	B	1/2" Tube	S/S	Whitey SS-45S8	U30						on hand		$62
39	CO	36	B	1/4" Tube	S/S	Swagelok SS-43S4	N7						on hand		$62
40	CO	37	B	1/4" Tube	S/S	Swagelok SS-43S4	L6						on hand		$62
41	CO	38	B	1/4" Tube	S/S	Swagelok SS-43S4	G5						on hand		$62
42	CO	39	B	1/2" Tube	S/S	Swagelok SS-43S8	H6						on hand		$62
43	CO	40	B	1/4" Tube	S/S	Swagelok SS-43S4	D7						on hand		$62
44	CO	41	B	1/4" Tube	S/S	Swagelok SS-43S4	T31						on hand		$62
45	CO	42	C	3/4" Pipe	S/S	Swagelok SS-CHF12-1/3	Y6				7/10/2007	7/17/2007	received		
46	CO	43A	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	W18				7/10/2007	7/17/2007	received	$11	
47	CO	43B	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	W18				7/24/2007		received		
48	CO	44	B	1/4" Tube	S/S	Swagelok SS-43S4	J11						on hand		
49	CO	45	B	1/4" Tube	S/S	Swagelok SS-43S4	J11						on hand		
50	CO	46	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	Y24						received		

	Valve List for Fractional Crystallization Test Facility														
Line #	Sys	Valve No.	Type	Size	Material	Vendor, Model No., and Other Information	P&ID Loc	Date Walkdown	Date Oper. Verification	Comments	Date Order Placed	Delivery Date	Status	Cost	Cost Avoided
51	CO	47	B	1/2" Tube	S/S	Swagelok SS-43S8	U7						on hand		
52	CO	48	B	1/4" Tube	S/S	Swagelok SS-43S4	D4						on hand		
53	CO	49	T	1/2" Tube	S/S	Swagelok SS-1RS8	R29								
54	CO	50	B	1/4" Tube	S/S	Swagelok SS-43S4	G34								
55	CO	51	B	1/4" Tube	S/S	Swagelok SS-43S4	G34								
56	CO	52	B	1/4" Tube	S/S	Swagelok SS-43S4	M35								
57	CO	53	B	1/2" Pipe	S/S	Parker 494F 316 w/tube adapters	M35								
58	CO	54	B	1/4" Tube	S/S	Swagelok SS-43S4	G34								
59	CO	55	B	1/4" Tube	S/S	Swagelok SS-43S4	M35								
60	CW	1	T	3" Pipe	Brass	Brass Gate	E17						on hand		$250
61	CW	2	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	B23				7/17/2007	7/23/2007	received	$89	
62	CW	3	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	C23				7/17/2007	7/23/2007	received	$89	
63	CW	4	T	1/2" Pipe	Brass	Gate Valve Stockham 200S 400 OWG	H19						on hand	$7	
64	CW	5	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	S9				7/10/2007	7/17/2007	received	$7	
65	CW	6	T	1/2" Tube	S/S	Swagelok SS-1RS8	T10						on hand		
66	CW	7	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	S11				7/10/2007	7/17/2007	received	$7	
67	CW	8	T	1/2" Tube	S/S	Swagelok SS-1RS8	U11						on hand		
68	CW	9	B	3/4" Tube	S/S	Whitey SS-45S10	R11						on hand		
69	CW	10	B	1/2" Tube	S/S	Whitey SS-45S8	R10						on hand		
70	CW	11	B	3/4" Tube	S/S	Whitey SS-45S10	Q11						on hand		
71	FR	1A	B	3/4" Pipe	CPVC	3/4" CPVC Ball Valve McMaster Carr #4724K82	AB20				7/10/2007	7/17/2007	received	$8	
72	FR	1B	B	3/4" Pipe	CPVC	3/4" CPVC Ball Valve McMaster Carr #4724K82	AB12				7/10/2007	7/17/2007	received	$8	
73	FR	1C	B	3/4" Pipe	CPVC	3/4" CPVC Ball Valve McMaster Carr #4724K82	AB14				7/10/2007	7/17/2007	received	$8	
74	FR	1D	B	3/4" Pipe	CPVC	3/4" CPVC Ball Valve McMaster Carr #4724K82	AB17				7/10/2007	7/17/2007	received	$8	
75	FR	2	B	3/4" Pipe	CPVC	3/4" CPVC Ball Valve Georg Fischer 163-346-303	AB8				7/10/2007	7/17/2007	received	$8	
76	FR	3	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AC8				7/10/2007	7/17/2007	received	$7	
77	FR	4	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AB7				7/10/2007	7/17/2007	received	$7	
78	FR	5	T	1/2" Pipe	SS	Parker 494F 316	V7						on hand	$200	
79	FR	6A	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AB20				7/10/2007	7/17/2007	received	$25	
80	FR	6B	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AB12				7/10/2007	7/17/2007	received	$25	
81	FR	6C	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AB15				7/10/2007	7/17/2007	received	$25	
82	FR	6D	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AB18				7/10/2007	7/17/2007	received	$25	
83	FR	6E	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AB22				7/10/2007	7/17/2007	received	$25	
84	FR	7	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	AB10				7/10/2007	7/17/2007	received	$25	
85	FR	8	B	3/4" Pipe	CPVC	3/4" CPVC Ball Valve McMaster Carr #4724K82	AA10				7/10/2007	7/17/2007	received	$7	
86	FR	9	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	X9				7/10/2007	7/17/2007	received	$25	
87	FR	10	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	Y8				7/10/2007	7/17/2007	received	$7	
88	FR	11A	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	Y20				7/10/2007	7/17/2007	received	$25	
89	FR	11B	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	Y11				7/10/2007	7/17/2007	received	$25	
90	FR	11C	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	Y14				7/10/2007	7/17/2007	received	$25	
91	FR	11D	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	Y17				7/10/2007	7/17/2007	received	$25	
92	FR	11E	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	Y22				7/10/2007	7/17/2007	received	$25	
93	FR	12A	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AA20				7/10/2007	7/17/2007	received	$7	
94	FR	12B	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AA12				7/10/2007	7/17/2007	received	$7	
95	FR	12C	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AA15				7/10/2007	7/17/2007	received	$7	
96	FR	12D	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AA18				7/10/2007	7/17/2007	received	$7	
97	FR	12E	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AA23				7/10/2007	7/17/2007	received	$7	
98	FR	13	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	W7				7/10/2007	7/17/2007	received	$20	
99	FR	14	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	X10				7/10/2007	7/17/2007	received	$7	
100	FR	15	C	1/2" Pipe	POLYPROPYLENE	McMaster Carr #5492K53	V7				7/24/2007		received		

	Valve List for Fractional Crystallization Test Facility														
Line #	Sys	Valve No.	Type	Size	Material	Vendor, Model No., and Other Information	P&ID Loc	Date Walkdown	Date Oper. Verification	Comments	Date Order Placed	Delivery Date	Status	Cost	Cost Avoided
101	FR	16	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	AB21						received		
102	FR	17	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	AB9				7/10/2007	7/17/2007	received	$20	
103	FR	18	B	1" Pipe	CPVC	1" CPVC Ball Valve	W9								
104	FR	19	B	1" Pipe	CPVC	1" CPVC Ball Valve	AC11								
105	FR	20	B	1" Pipe	CPVC	1" CPVC Ball Valve	Y10								
106	PA	1	B	3/4" Tube	S/S	Whitey SS-45F10	Z10						on hand		$180
107	PA	2A	B	1/4" Tube	S/S	Swagelok SS-43S4							on hand		
108	PA	2B	B	1/4" Tube	S/S	Swagelok SS-44F4 w/tube adapters	S4						on hand		$62
109	PA	3	B	1/4" Tube	S/S	Swagelok SS-43S4	L38						on hand		
110	PA	4	T	1/4" Pipe	Brass	Part of centrifuge									
111	PD	1	B	2" Pipe	CPVC	2" CPVC Ball Valve McMaster Carr #4724K86	U36				7/10/2007	7/17/2007	received	$25	
112	PD	2	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	V36				7/10/2007	7/17/2007	received	$7	
113	PD	3	T	1" Pipe	S/S	316 SS Globe Valve McMaster #4742K15	S34				7/17/2007	7/23/2007	received	$89	
114	PD	4	T	1/4" Tube	S/S	Swagelok SS-1RS4	V31				6/15/2007	6/27/2007	received	$49	
115	PD	5	A/A (NC)	1/4" Tube	S/S	Assured Automation Mod. A26NRX08SCE3A	V31				7/23/2007		received	$319	
116	PD	6	T	3/8" Tube	S/S	Swagelok SS-1VS6	V31				6/18/2007	6/27/2007	received	$68	
117	PD	7	A/A (NO)	1/2" Pipe	S/S	Assured Automation Mod. C26NRX08SOE3A	R31				7/23/2007		received	$403	
118	PD	8	A/A 3W	1/2" Pipe	S/S	Assured Automation Mod. C33DAX08S1E3A	L32				7/23/2007		received	$417	
119	PD	10	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	P31						on hand		$200
120	PD	11	B3W	3/8" Tube	S/S	Swagelok SS-44XS6	S32						on hand		$62
121	PD	12	B 3W	3/8" Tube	S/S	Swagelok SS-44XS6	T31						on hand		
122	PD	13B	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	X17				7/10/2007	7/17/2007	received	$7	
123	PD	13C	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	X17				7/10/2007	7/17/2007	received	$7	
124	PD	13D	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	Y18				7/10/2007	7/17/2007	received	$7	
125	PD	14	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	U34						received		
126	PD	15	B	1" Pipe	CPVC	1" CPVC Ball Valve McMaster Carr #4724K83	U33						received		
127	PD	17	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81							received		
128	RW	1	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	U27				7/10/2007	7/17/2007	received	$20	
129	RW	2	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	V26				7/10/2007	7/17/2007	received	$7	
130	RW	3	T	1" Pipe	S/S	316 SS Globe Valve McMaster #4742K15	U25				7/17/2007	7/23/2007	received	$89	
131	RW	4	T	3/8" Tube	S/S	Swagelok SS-1VS6	U24				6/18/2007	6/27/2007	received	$68	
132	RW	5	A/A 3W	1/2" Pipe	S/S	Assured Automation Mod. C33DAX08S1E3A	J33				7/23/2007		received	$417	
133	RW	6	A/A 3W	3/4" Pipe	S/S	Assured Automation Mod. D33DAX08S1E3A	L33				7/23/2007		received	$462	
134	RW	7	B 3W	3/4" Pipe	CPVC	McMaster Carr #4697K42	N28				7/10/2007	7/17/2007	received	$107	
135	RW	8	B3W	1/4" Tube	S/S	Swagelok SS-43XS4	T26						on hand		
136	SL	1	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	V9				7/10/2007	7/17/2007	received	$25	
137	SL	2	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	V9				7/10/2007	7/17/2007	received	$7	
138	SL	3	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	F34				7/10/2007	7/17/2007	received	$7	
139	SL	4	B 3W	1 1/2" Pipe	CPVC	McMaster Carr #4697K44	W8				7/10/2007	7/17/2007	received	$233	
140	SL	5	P	1 1/2" Pipe	Viton	Red Valve Series 70	V8				6/15/2007	7/11/2007	on hand	$499	
141	SL	6	A/A 3W	1" Pipe	S/S	Assured Automation Mod. E33DAX08S1E3A	G36				7/23/2007		received	$547	
142	SL	7	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	W9				7/10/2007	7/17/2007	received	$7	
143	SL	8			S/S	Custom Design and SRNL Manufacture	D4								
144	SL	9	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	X6				7/10/2007	7/17/2007	received	$7	
145	SL	10	B	1" Pipe	CPVC	1" CPVC Ball Valve McMaster Carr #4724K83	W6				7/10/2007	7/17/2007	received	$7	
146	SL	11	B	1/2" Tube	S/S	Swagelok SS-45S8	P6						on hand		
147	SL	12A	AT	1/4" Tube	S/S	Brooks flow controller Model # 58501A1BT342BEA	A18				9/10/2007	9/25/2007	received		
148	SL	12B	B	1/2" Tube	S/S	Swagelok SS-45S8							on hand		
149	SL	12M	T	1/2" Tube	S/S	Hoke 2112G8V	B18						on hand		
150	SL	13	B	1/4" Tube	S/S	Swagelok SS-43S4	P8						on hand		$62

	Valve List for Fractional Crystallization Test Facility														
Line #	Sys	Valve No.	Type	Size	Material	Vendor, Model No., and Other Information	P&ID Loc	Date Walkdown	Date Oper. Verification	Comments	Date Order Placed	Delivery Date	Status	Cost	Cost Avoided
151	SL	14	B	1/4" Tube	S/S	Swagelok SS-43S4	M7						on hand		$62
152	SL	15	B	1/2" Tube	S/S	Swagelok SS-45S8	H7						on hand		$250
153	SL	16	B	1/4" Tube	S/S	Swagelok SS-43S4	D8						on hand		$62
154	SL	17	B	1" Pipe	CPVC	1" CPVC Ball Valve Georg Fischer	G35						on hand		
155	SL	18B	B	3/4" Tube	S/S	Swagelok SS-45S12	E18						on hand		
156	SL	18T	B	1/2" Tube	S/S		E19						on hand		
157	SL	19	B	1/2" Tube	S/S	Swagelok SS-45S8	E3						on hand		
158	SL	20	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81									
159	SL	21	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81									
160	ST	1	B	1" Pipe	Brass	Brass Ball, Installed	AB2						Installed		
161	ST	2	B	1" Pipe	Brass	Brass Ball, Installed	AB4				7/10/2007		received	$14	
162	ST	3	AT	1" Pipe	S/S	Schubert & Salzer 8021/025VGE106M--91-1ZR-S-2	V3				6/28/2007		received	$2,058	
163	ST	4	T	1" Pipe	Brass	Stockham Fig. B-22T Brass 150S 300CWP	K1				on hand		installed		$100
164	ST	5	T	1" Pipe	Brass	Stockham Fig. B-22T Brass 150S 300CWP	K1				on hand		installed		$100
165	ST	6	B	3/8" Tube	S/S	Swagelok SS-43S6	P5						on hand		$64
166	ST	7	B	1/2" Tube	S/S	Swagelok SS-43S8	Q5						on hand		$64
167	ST	8	T	1/2" Tube	S/S	Hoke 2112G8V	L11				6/18/2007	6/27/2007	received	$68	
168	ST	9	B	1/2" Tube	S/S	Swagelok SS-43S8	S7						on hand		$64
169	ST	10	T	1/4" Pipe	S/S	Hoke 2112F4Y	L35						on hand		
170	ST	11	T	1/2" Pipe	BRASS	Gate Valve Stockham 200S 400 OWG	AA5						on hand		
171	ST	12	T	1/4" Pipe	S/S	Hoke 2112F4Y	M2						on hand		
172	SW	1	B	1 1/2" Pipe	CPVC	1-1/2" CPVC Ball Valve McMaster Carr #4724K85	U22				7/10/2007	7/17/2007	received	$20	
173	SW	2	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	V21				7/10/2007	7/17/2007	received	$7	
174	SW	3	T	1" Pipe	S/S	316 SS Globe Valve McMaster #4742K15	U20				7/17/2007	7/23/2007	received	$89	
175	SW	4	T	1/4" Tube	S/S	Swagelok SS-1RS4	Q19				6/15/2007	6/27/2007	received	$49	
176	SW	5	A/A (NC)	1/4" Tube	S/S	Assured Automation Mod. A26NRX08SCE3A	Q19				7/23/2007		received	$319	
177	SW	6	T	3/8" Tube	S/S	Swagelok SS-1VS6	Q18				6/18/2007	6/27/2007	received	$68	
178	SW	7	T	1/2" Pipe	SS	Parker 494F 316	M17						on hand		$200
179	SW	8	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81	T8				7/10/2007	7/17/2007	received	$7	
180	SW	9	A/A 3W	3/4" Pipe	S/S	Assured Automation Mod. D33DAX08S1E3A	M34				7/23/2007		received	$462	
181	SW	10	B3W	3/4" Pipe	CPVC	McMaster Carr #4697K42	N23						received	$235	
182	SW	11	B3W	3/8" Tube	S/S	Swagelok SS-44XS6	T20						on hand		$62
183	SW	12	B 3W	1/2" Pipe	CPVC	McMaster Carr #4697K41	M18				7/10/2007	7/17/2007	received	$95	
184	SW	13	B 3W	1/2" Pipe	CPVC	McMaster Carr #4697K41	N19				7/10/2007	7/17/2007	received	$95	
185	SW	14	A/A 3W	1/2" Pipe	S/S	Assured Automation Mod. C33DAX08S1E3A	H34				7/23/2007		received	$417	
186	SW	15	B	1/2" Pipe	CPVC	1/2" CPVC Ball Valve McMaster Carr #4724K81					7/10/2007	7/17/2007	received	$7	

APPENDIX 8.7

PILOT DAS INDICATIONS

**(Consisting of 5 pages
Including coversheet)**

DAS Ch. #	Cabinet Conn. Label	Funct-ion	Loop ID	TR#	Quantity Measured	Measuring Device	Local /DAS	Range	Oper. Verified	Low Alarm	High Alarm	Tolerance	Purch Resp*	Vendor, Model No, and Other Information
0	T1	TE	CO01	TR-03858	Condensate Tank Temp.	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
1	T2	TE	CW01	TR-03857	Condenser Cooling Water Inlet Temp.	Thermocouple	DAS	32-212 F	MR			3.6 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
2	T3	TE	CW03	TR-03846	Condenser Cooling Water Outlet Temp.	Thermocouple	DAS	32-212 F	MR		XXX	3.6 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
3	T4	TE	CO10	TR-03908	Temp.of Condensate Feed to Product Disolver	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
4	T5	TE	CW06	TR-03899	Jet Pump 1st. Stage Cooling Water Temp. outlet	Thermocouple	DAS	32-212 F	MR		XXX	3.6 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
5	T6	TE	CW08	TR-03901	Jet Pump 2nd. Stage Cooling Water Temp. outlet	Thermocouple	DAS	32-212 F	MR		XXX	3.6 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
6	T7	TE	CW10	TR-03856	Seal Water Temperature	Thermocouple	DAS	32-212 F	MR		XXX	3.6 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
7	T8	TE	FR03	TR-03851	Feed Temperature	Thermocouple	DAS	32-212 F	MR			3.6 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
8	T9	TE	PD01	TR-03862	Product Dissolver Tank Temp.	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
9	T10	TE	RW01	TR-03906	Recycle Wash Tank Temp.	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
10	T11	TE	RW05	TR-03843	Recycle Wash Centrifuge Feed Temperature	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
11	T12	TE	SL12	TR-03845	Slurry Temp. to Centrifuge	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
12	T13	TE	ST02	TR-03904	Steam Header Temperature	Thermocouple	DAS	32-392 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
13	T14	TE	SW01	TR-03852	Spent Wash Tank Temp.	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
14	T15	TW	SL04	TR-03861	Crystallizer Vessel Temperature	Thermocouple	DAS	32-212 F	MR	???	XXX	1.8 +/- F	JC	1/4", 12"L, Type E, Omega # GEQSS-14(G)-12
15	T16	TW	ST04	TR-03905	Steam Temperature after Desuperheater	Thermocouple	DAS	32-212 F	MR		XXX	1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
16	T17	TW	ST05	TR-03851	Condensate Temp. at Reboiler exit	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
17	T18	TE	PD05	03848 destro	Product Dissolver Centrifuge Feed Temperature	Thermocouple	DAS	32-212 F	MR			1.8 +/- F	JC	1/16", 12"L, Type E, Omega # GEQSS-116(G)-12
18	T19				Spare TC	Thermocouple								
19	T20				Spare TC	Thermocouple								
20	T21				Spare TC	Thermocouple								
21	T22				Spare TC	Thermocouple								
22	A01	CT	SL10	TR-03891	Slurry Conductivity in Drawoff Line	Transmitter	DAS	0-2 S/cm	MR, MDF			0	JS	Omega CDTX-45T1 Conductivity analyzer
23	A02	DT	PD06	TR-03490	Product Dissolver Density (Sp.Gr.)	Diff. Press Trans	DAS	0-100 in wc	MR	XXX	XXX	0	ZQ	Rosemount
24	A03	~~DT~~	~~SL05~~	~~TR-03493~~	~~Crystallizer Slurry Density~~	~~Diff. Press Trans~~	~~DAS~~	~~0-100 in wc~~				~~0.5% +/- FS~~	BAD CHANNEL	~~Rosemount~~
25	A04	LT	SL06	TR-03691	Crystallizer Vessel Level	Diff. Press Trans	DAS	0-200 in wc	MR	0%	100%	0.5% +/- FS	ZQ	Rosemount
26	A05	PDT	SL08	TR-03106	Pressure Drop across Filter Pads	Diff. Press Trans	DAS	0-30 in wc	MR		XXX	0.5% +/- FS	ZQ	Rosemount
27	A06	TDW	SL02	TR-03889	Slurry Temp. rise across Reboiler	Diff. T/couple	DAS	0-20 C	MR, TJS		XXX	+/- 0.25 C	TS	Omega 5TC-GG-E-20-72
28	A07	FT	ST01	TR-03688	Reboiler Steam Flow Rate	DP across orifice	DAS	0-30 in wc	MR			0.5% +/- FS	ZQ	Rosemount
29	A08	FT	CO11	TR-03563	Condensate to Product Dissolver Flow Rate	Magnetic FM	DAS	0-2 gpm	MR			4% of reading	JC	0
30	A09	FT	CO18	TR-03677	Condensate Flow to HLW Receipt Tank	Magnetic FM	DAS	0-2 gpm	MR			4% of reading	JC	0
31	A10	FT	FR01	TR-03678	Feed Flow Rate	Magnetic FM	DAS	0-2 gpm	MR			4% of reading	JC	0
32	A11	FT	PD09	TR-03276	Product Dissolver Tank Purge Flow	Magnetic FM	DAS	0-2 gpm	MR			4% of reading	JC	0
33	A12	FT	PD10	TR-03704	Product Dissolver Centrifuge Feed Flow Rate	Magnetic FM	DAS	0-5 gpm	MR			4% of reading	JC	0
34	A13	FT	SW06	TR-03721	Spent Wash to HLW Tank Flow Rate	Magnetic FM	DAS	0-2 gpm	MR			4% of reading	JC	TR-03721
35	A14	FT	SW09	TR-03705	Spent Wash Flow to Crystallizer	Magnetic FM	DAS	0-2 gpm	MR			4% of reading	JC	TR-03705
36	A15	PT	CO12	TR-03717	Interstage Jet Pump Pressure	Press Transd	DAS	0-15 psia	MR		XXX	0.5% +/- FS	JC	`

DAS Ch. #	Cabinet Conn. Label	Funct-ion	Loop ID	TR#	Quantity Measured	Measuring Device	Local /DAS	Range	Oper. Verified	Low Alarm	High Alarm	Tolerance	Purch Resp*	Vendor, Model No, and Other Information	
37	A16	PT	SL07	TR-03885	Crystallizer Vessel Pressure	Pressure Trans	DAS	0-15 psia	MR	XXX	XXX	0.5% +/- FS	ZQ	Omega, PX4200-30VACI	
38	A17	PE	ST06	TR-03883	Reboiler Shell Steam Pressure Element	Pressure Transd	DAS	0-15 psia	MR	XXX	XXX	0.5% +/- FS	JG	Omega, PX4200-30VACI	
39	A18	LC	PD07	N/A	Product Dissolver Tank Level Controller	Ultrasonic	DAS	0.3 - 6 ft	MR	XXX	XXX	0.125 in	TS	Omega LVCN-51	
40	A19	LE	RW07	TR-03918	Recycle Wash Tank Level	Ultrasonic	DAS	0.3 - 6 ft	MR	XXX	XXX	0.125 in	TS	Omega LVU91	
41	A20	LC	SW07	N/A	Spent Wash Tank Level Controller	0	DAS	0.3 - 6 ft	MR	XXX	XXX	0.125 in	TS	Omega LVCN-51	
42	A21	FT	SL03	TR-03892	Circulation Flow Rate	Ultrasonic FM	DAS	0-1000 gpm	MR	XXX		2 %rdg+0.2 fps	ZQ	GE TransPort PT878	
43	A22	CT	CO02	TR-03884	Condensate Conductivity Transmitter	0	DAS		MR, MDF			2%rdg+6uS/cm	TS	Omega CDCN-91AC	
44	A23	IT	SL01	N/A	Circulation Pump Current	0	DAS	0-20 amps	MR		**High Positive Rate of Change**			0	
45	A24	ST	SL01	N/A	Circulation Pump Speed	0	DAS	0-3550 rpm	MR			0	MR	0	
46	A25	FT	SL14	TR-03601	Draw Off Return Flow Rate	Magnetic FM	DAS	0-100 gpm	MR	XXX		4% of reading	JC	TR-03601	
47	A26	FT	CW02	TR-03687	Condenser Cooling Water Flow Rate	Magnetic FM	DAS	0-200 gpm	MR			4% of reading	MR	0	
48	A27	DT	SL05	TR-03493	Crystallizer Slurry Density	Diff. Press Trans	DAS	0-100 in wc	TS, MR	XXX	XXX	0.5% +/- FS	ZQ	Rosemount	
49	A28	FT	SL15	CDS-4714	Draw Off Loop Flow Rate (Ultrasonic)	Ultrasonic FM	DAS	0-60 gpm	MR, MDF			1.5%rdg+0.15fp	MF	Controllotron System 990	
50	A29	FT	SL12	N/A	SL12 Mass Flowmeter	Mass Flow Meter	DAS	0-20 SLPM	MR, MDF				MR	Brooks flow controller Model # 58501A1BT342BEA	
51	A30				Spare 4-20 mA Channel										
52	A31				Spare 4-20 mA Channel										
53	A32				Spare 4-20 mA Channel										
54	A33				Spare 4-20 mA Channel										
55	A34				Spare 4-20 mA Channel										
56	A35				Spare 4-20 mA Channel										
57	A36				Spare 4-20 mA Channel										
58	A37				Spare 4-20 mA Channel										
59	A38				Spare 4-20 mA Channel										
60	A39	SV	PD05		Valve Indication for PD05	Switch	DAS	On/Off	MR						
61	A40	SV	SW05		Valve Indication for SW05	Switch	DAS	On/Off	MR						
62	A41	SV	PD07		Valve Indication for PD07	Switch	DAS	On/Off	MR						
63	A42	SV	CO08		Valve Indication for CO08	Switch	DAS	On/Off	MR						
64	A43	SV	CO33		Valve Indication for CO33	Switch	DAS	On/Off	MR						
65	A44	SV	PD08		Valve Indication for PD08	Switch	DAS	On/Off	MR						
66	A45	SV	RW05		Valve Indication for RW05	Switch	DAS	On/Off	MR						
67	A46	SV	SW14		Valve Indication for SW14	Switch	DAS	On/Off	MR						
68	A47	SV	RW06		Valve Indication for RW06	Switch	DAS	On/Off	MR						
69	A48	SV	SW09		Valve Indication for SW09	Switch	DAS	On/Off	MR						
70	A49	SV	SL06		Valve Indication for SL06	Switch	DAS	On/Off	MR						
71	A50	LAH	ALL LAH		All LAH High Level Alarms	Float Switches	DAS	On/Off	MR		XXX				

DAS Ch. #	Cabinet Conn. Label	Funct-ion	Loop ID	TR#	Quantity Measured	Measuring Device	Local /DAS	Range	Oper. Verified	Low Alarm	High Alarm	Tolerance	Purch Resp*	Vendor, Model No, and Other Information
Analog Out														
AO-0	AO-0	SV	CO32		Output to SV CO32	Valve Position Input	DAS	4-20 mA	MR					
AO-1	AO-1	SV	SL12		Output to SV SL12	Valve Position Input	DAS	4-20 mA	MR					
AO-2	AO-2	SV	FR05		Output to VFD for Pump FR Pump 1	Speed Control	DAS	4-20 mA	MR					
AO-3	AO-3				Spare Analog Output Channel									
AO-4	AO-4				Spare Analog Output Channel									
AO-5	AO-5				Spare Analog Output Channel									
AO-6	AO-6				Spare Analog Output Channel									
AO-7	AO-7				Spare Analog Output Channel									
Digital Input/Output														
DIO-0	DIO-0				FR05 Power/Leeson VFD 174917	Power Supply Switch	DAS	NO Contact	MR					
DIO-1	DIO-1				Spare Digital Input/Output Channel									
DIO-2	DIO-2				Spare Digital Input/Output Channel									
DIO-3	DIO-3				Spare Digital Input/Output Channel									
DIO-4	DIO-4				Spare Digital Input/Output Channel									
DIO-5	DIO-5				Spare Digital Input/Output Channel									
DIO-6	DIO-6				Spare Digital Input/Output Channel									
DIO-7	DIO-7				Spare Digital Input/Output Channel									
Miscellaneous Control Loops														
Valve C005									MR					
Valve C032									MR					
Valve SL12														
Valve SW05									MR					
Valve PD05									MR					
Loop ST06														
PD Heater System									DF, MDF					
SW Heater System									DF, MDF					
RW Heater System									DF, MDF					
Steam Generator Problems									BG, TJS					

DAS Ch. #	Cabinet Conn. Label	Funct-ion	Loop ID	TR#	Quantity Measured	Measuring Device	Local /DAS	Range	Oper. Verified	Low Alarm	High Alarm	Tolerance	Purch Resp*	Vendor, Model No, and Other Information
					LOCAL INSTRUMENTS									
		FI	CO04	TR-00122	Condensate Flow Rate to Crystallizer Spray Ring	Rotameter	Local	0-5 gpm				+/- 0.35 gpm	SS	0
		FI	CO05	TR-20268	Condensate Flow to Crystallizer Lower Demister	Rotameter	Local	0-2 gpm				+/- 0.15 gpm	SS	0
		FI	CO06	TR-20269	Condensate Flow to Crystallizer Upper Demister	Rotameter	Local	0-2 gpm				+/- 0.15 gpm	SS	0
		FI	CO07	#REF!	Condensate Flow for Desuperheater	Rotameter	Local	0-20 gph				+/- 0.4 gph	SS	Fischer & Porter 10A6132M
		FI	CO09	TR-03887	Centrifuge Rind Wash Water Totalizer		Local	0.22-22 gpm				2.5% of rdg	TS	Omega FTB80007A
		FI	CO13	TR-03510	Condensate Purge Flow to Crystallizer Nozzle #10	Rotameter	Local	0-2 gph				4% of reading	SS	
		FI	CO14	TR-03658	Condensate Purge Flow to Crystallizer Nozzle #6	Rotameter	Local	0-2 gph				4% of reading	SS	
		FI	CO15	TR-03878	Condensate Purge Flow to Crystallizer Nozzle #7	Rotameter	Local	0-2 gph				4% of reading	SS	
		FI	CO16	TR-03879	Condensate Purge Flow to Crystallizer Nozzle #8	Rotameter	Local	0-2 gph				4% of reading	SS	
		LC	CO19	N/A	Condensate Tank Level Control	0	0	0				0	Omega LV611-P	Omega LV611-P
		FI	CO20	TR-03880	Condensate Purge Flow to PD Density Transmitter	Rotameter	Local	0-2 gph				4% of reading	SS	
		FI	CO21	TR-03881	Condensate Purge Flow to PD Density Transmitter	Rotameter	Local	0-2 gph				4% of reading	SS	
		FI	CW05	N/A	Jet Pump Cooling Water 1st Stage Flow Rate	Rotameter	Local	0-20 gpm				4% of reading	SS	
		FI	CW07	N/A	Jet Pump Cooling Water 2nd Stage Flow Rate	Rotameter	Local	0-20 gpm				4% of reading	SS	
		LAH	RW06	N/A	Recycle Wash Tank High Level Alarm	Float Switch	Local	0				0	JC	McMaster Carr #46515K41 Polypropylene
		HS	PD12	N/A	PD Filter Backwash Switch	N/A	0	0				0	TS	0
		HS	SW05	N/A	Sample Switch	Switch	Local	N/A				N/A	TS	0
		LAH	CO03	N/A	Condensate Tank High Level Alarm	Float Switch	Local					0	JC	McMaster Carr #46515K41 Polypropylene
		LAH	FR04A	N/A	Feed Tank A High Level Alarm	Float Switch	Local	0				0	JC	McMaster Carr #46515K41 Polypropylene
		LAH	FR04B	N/A	Feed/LAW Tank B High Level Alarm	Float Switch	Local	0				0	JC	McMaster Carr #46515K41 Polypropylene
		LAH	FR04C	N/A	Feed/LAW Tank C High Level Alarm	Float Switch	Local	0				0	JC	McMaster Carr #46515K41 Polypropylene
		LSH	CO08	N/A	Reboiler Condensate receiver level	Float Switch	Local					0	JG	Dwyer/WE Anderson, F7-HPS-21
		LSL	CO08	N/A	Reboiler Condensate receiver level	Float Switch	Local					0	JG	Dwyer/WE Anderson, F7-HPS-21
		PI	CO17	N/A	Condensate Header Pressure	Pressure Gage	Local	0-100 psig				0	JC	Moore Products Co. #3994
		PI	CO22	N/A	Bottom Spray Nozzle supply pressure	Pressure Gage	Local	Vac-60 psig				2% of reading	JC	McMaster Carr #38545K16
		PI	CO23	N/A	Top Spray Nozzle supply pressure	Pressure Gage	Local	Vac-60 psig				2% of reading	JC	McMaster Carr #38545K16
		PI	CW09	N/A	Circulation Pump seal water pressure	Pressure Gage	Local	0-100 psig				0	JC	Glycerin-Filled McMaster Carr #4088K511
		PI	FR02	N/A	Feed Pressure	Pressure Gage	Local	0-30 psig				0	JC	Glycerin-Filled McMaster Carr #4088K511
		PI	PD04	N/A	Product Dissolver Filter Inlet Pressure	Pressure Gage	Local	0-60 psig				0	JC	Glycerin-Filled McMaster Carr #3813K7
		PI	PD11	N/A	Product Dissolver Filtrate Pressure	Pressure Gage	Local	0-30 psig				0	JC	Glycerin-Filled McMaster Carr #3813K7
		PI	RW04	N/A	Recycle Wash Pressure	Pressure Gage	Local	0-60 psig				0	JC	Glycerin-Filled McMaster Carr #3813K7
		PI	SL11	N/A	Draw Off Line Pressure	Pressure Gage	Local	vac-30 psig				1.5% FS	ZQ	hok 25-400 30"Hg/30 psig + McMaster isolator 4334
		PI	ST03	TR-03886	Steam Header Pressure	Pressure Gage	Local	0-160 psig				0	JC	Glycerin-Filled McMaster Carr #4088K511
		PI	ST07	N/A	Jet Pump Steam Pressure for 2nd. Stage	Pressure Gage	Local	0-160 psig				0	JC	Glycerin-Filled McMaster Carr #4088K511
		PI	ST08	N/A	Jet Pump Steam Pressure for 1st. Stage	Pressure Gage	Local	0-160 psig				0	JC	Glycerin-Filled McMaster Carr #4088K511
		PI	SW04	N/A	Spent Wash Pressure	Pressure Gage	Local	0-60 psig				0	JC	Glycerin-Filled McMaster Carr #3813K7
		SC	FR05	N/A	Feed/Receipt Pump 1 VFD	VFD	Local	0				0	JC	0

APPENDIX 8.8

PILOT EQUIPMENT LIST

**(Consisting of 4 pages
Including coversheet)**

Equipment List for Fractional Crystallization Test Facility												
Sys	No.	Item Description	Specifications	Vendor, Model No., and Other Information	Date Walkdown	Date Oper. Verification	Cost	Cost Avoided	Date Order Placed	Delivery Date	Resp*	Status
CO	0	**Condensate System**										
CO	1	Primary Condenser	1,000,000 Btu/hr	PO # is AC60362						08/31/07	ZQ	installed
CO	2	Steam Jet Vacuum Pump	0.8 psia									existing
CO	3	Condensate Tank	360 gal LDPE	National Tank OP0360			$235			08/20/07	SS	received
CO	4	Condensate Pump 1		Dynaflow Engineering MT5003-P3T6 Regenerative turbine pump, polypropylene			$1,948				TS	received
CO	5	Condensate Pump 2		ITT B&G, Type B, Series B, 616B, 2.5 gpm			$1,595		06/18/07		JG	installed
CO	6	Condensate Tank F	1500 Gallon LDPE								JC	on hand
CO	7	Condensate Tank G	1000 Gallon LDPE								JC	on hand
CO	8	Steam Generators Feed Water Filter									JC	on hand
CO	9	Backpressure Regulator	0-50 psig	Fisher Model 98H-33 (1" cast iron body, NPT conn, metal diaph, 25-75 psig spring)			$450		07/30/07		TS	received
CO	10	Desuperheater Spray Nozzle		Spraying Systems, 1/4AX-SS1-5W			$55		06/18/07	06/21/07	JG	received
CO	11	Condensate Receiver		749-A shop modified inlets & outlets							JC	installed
CO	12	Bag Filter 200	200 microns									
CW	0	**Cooling Water**										
CW	1	CW Pump 1 (Seal water Pump)		Teel 2P372								on hand
CW	2	Seal Water tank										on hand
CW	3	Seal Water Filter	25 then 1 micron	Pentek Cartridge DGD-2501-20 in Cole-Parmer C-01507-77 "Big Blue" housing							TS	on hand
FR	0	**Feed/Receipt System**										
FR	1	Feed Tank A	1500 gal LDPE								JC	on hand
FR	2	Feed/LAW Tank B	2500 gal LDPE	National Tank DCB2500-90 LHDPE Cone Bottom			$1,573			08/20/07	SS	received
FR	3	Feed/LAW Tank C	2500 gal LDPE	National Tank DCB2500-90 LHDPE Cone Bottom			$1,573			08/20/07	SS	received
FR	4	Feed/LAW Tank D	2500 gal LDPE	National Tank DCB2500-90 LHDPE Cone Bottom			$1,573			08/20/07	SS	received
FR	5	HLW Tank E	1500 gal LDPE								JC	on hand
FR	6	Feed/Receipt Pump 1	2 gpm	Teel, 1P801B centrifugal, 3/4 hp, 208 v 3P, 6 gpm @ 60' hd							JC	on hand
FR	7	Feed/Receipt Pump 2	50 gpm	Teel, 2P392 centrifugal 3 hp, 208v, 3P, 70gpm@60' hd, or Wilden air driven double diaphragm pump P8/SAPP/UV/VT/VT/044, 70 gpm with about 40 scfm air							JC	on hand
FR	8	Mixer for HLW Tank E		Lightnin Mod. 1620-50 120v 8a							JC	on hand
FR	9	Blade for HLW Tank Mixer		Lightnin, A-310							JC	on hand
MC	0	**Miscellaneous**										
MC	1	Crystalizer to Cir. Pump Piping	6" schd 10 S/S	749-A machine shop fabricated							TS	received
MC	2	Cir. Pump to Reboiler Piping	6" schd 10 S/S	749-A machine shop fabricated							TS	installed
MC	3	Reboiler to Crystallizer Piping	6" schd 10 S/S	749-A machine shop fabricated							TS	installed
MC	4	Crystallizer to Condenser Piping	8" schd 10 S/S	749-A machine shop fabricated							TS	installed
MC	5	6" Reboiler Steam Supply Piping	6" schd 10 S/S	749-A machine shop fabricated							JC	received
MC	6	Sec. Cont. for Crystallizer	1500 gallons				$3,420				SS	installed
MC	7	Sec. Cont. for Outside Cond. Area	1800 gallons								SS	on hand
MC	8	Sec. Cont. for Feed Tank Area	3000 gallons	Existing Secondary Cont. 25' x 15' x 1' 2800 Gal.				$6,000			JC	on hand

Equipment List for Fractional Crystallization Test Facility												
Sys	No.	Item Description	Specifications	Vendor, Model No., and Other Information	Date Walkdown	Date Oper. Verification	Cost	Cost Avoided	Date Order Placed	Delivery Date	Resp*	Status
MC	9	Sec. Cont. for Process Tanks & Ce	750 gallons				$2,870				SS	received
MC	10	Thermocouple Extension Wire	Type E						06/27/07		JC	received
MC	11	Early Feed Simulant (no Cs & Cr)	10,000 gal								MW	ordered
MC	12	Sodium dichromate Solution	500 gal PP								MW	ordered
MC	13	Cesium nitrate	25 kg								MW	ordered
MC	14	Microscope Parts for PLM		McCrome Associates, McCrome will be on site in August to train SRNL					06/15/07		MW	received
MC	15	Condenser Support Frame	Per DWG	749-A machine shop will fabricate					07/12/07		DF	frabricated
MC	16	Platform Supports	Per DWG	749-A machine shop will fabricate					07/12/07		DF	installed
MC	17	Springs for Axial Pump support	K =544 lb/in	McMaster-Carr 9624K33			$44				TS	received
MC	18	Flange and Seal for Press Relief	DWG	749-A machine shop will fabricate							TS	received
MC	19	CPVC sweeps for drawoff line	5D Bends				$1,000				TS	received
MC	20	Gaskets for piping connections	Various sizes								TS	received
MC	21	Spare gasket for crystallizer	24" Flexatalic						09/10/07		JC	received
MC	22	Instrument air dryer for centrifuge seals									MR	on hand
PA	0	**Plant Air System**										
PA	1	Pressure Regulator for Centrifuge	35 SCFH @ 5 psig								JC	on hand
PA	2	Air Dryer									MR	on hand
PD	0	**Product Dissolver System**										
PD	1	Product Dissolver Tank	500 gal PP	Chemtainer, TC5264CB / National Tank DOPT0500			$1,060			09/07/07	SS	received
PD	2	PD Tank Stand		Chemtainer, CK-CM5264CB Stand							JC	on hand
PD	3	PD Tank Baffles									JC	on hand
PD	4	Mixer for PD Tank									JC	on hand
PD	5	Blade for PD Tank Mixer		Lightnin, A-310							JC	on hand
PD	6	Product Dissolver Pump		Teel, 2P392 centrifugal, 3 hp, 208 v 3P, 28 gpm @ 70' hd & 70 gpm @ 60' hd							JC	on hand
PD	7	PD Electrical Heater		SRNL Fabricated ST-MDX5-9878							DF	on hand
PD	8	PD Crossflow Filter Tubes Only								10/05/07	TS	received
PD	9	PD Crossflow Filter Housing		Custom design, fabrication in 749-A complete, assembly in EDL							TS	installed
RW	0	**Recycle Wash System**										
RW	1	Recycle Wash Tank	500 gal PP	Chemtainer,TC5264CB (received cracked, can use on-hand polyethylene tank for fitup and shakedown. Can also use for operation at current planned temperatures)			$1,060			09/07/07	SS	received
RW	2	RW Tank Stand		Chemtainer, CK-CM5264CB Stand							JC	on hand
RW	3	Mixer for RW Tank		Lightnin Mod. 1620-50 120v 8a							JC	on hand
RW	4	Blade for RW Tank Mixer		Lightnin, A-310							JC	on hand
RW	5	Recycle Wash Pump		Teel, 1P701B centrifugal, 3/4 hp, 115 v 1P, 18 gpm @ 50' hd & 30 gpm @ 40' hd							JC	on hand
RW	6	RW Electrical Heater		SRNL Fabricated ST-MDX5-9878							DF	on hand

Equipment List for Fractional Crystallization Test Facility												
Sys	No.	Item Description	Specifications	Vendor, Model No., and Other Information	Date Walkdown	Date Oper. Verification	Cost	Cost Avoided	Date Order Placed	Delivery Date	Resp*	Status
SL	0	**Slurry System**										
SL	1	Crystallizer Vessel									Areva	installed
SL	2	Circulation Pump	6" Axial flow pump							10/03/07	Areva	received
SL	3	Reboiler									Areva	received
SL	4	Draw Off Pump							09/05/07	10/31/07	RL	received
SL	5	Centrifuge								10/10/07	Areva	received
SL	6	Motor Starter for Centrifuge		McMaster-Carr 6559K66 motor starter + 6559K52 enclosure + 9543K13 conduit adapters							TS	received
SL	7	Plug for 480 volt weld receptacle (2 rq'd)		Crouse-Hinds APJ10487 or equal (McMaster-Carr 7316K38)			$750				TS	received
SL	8	Motor Starter for Drawoff Pump		McMaster-Carr 6559K67 motor starter + 6559K52 enclosure + 9543K13 conduit adapters			$141				TS	received
ST	0	**Steam System**										
ST	1	Steam Generator 1 (150 kW)	Electrical Heaters	Electro-Steam, LB-150								installed
ST	2	Steam Generator 2 (240 kW)	Electrical Heaters	Electro-Steam, LB-240							MR	received
ST	3	Pressure Relief Valve for SG1										installed
ST	4	Pressure Relief Valve for SG2		Ready to be installed							MR	received
ST	5	Piping for steam supply	schd 40 pipe, 2000#	Purchase request issued to buyer					09/10/07		JC	received
SW	0	**Spent Wash System**										
SW	1	Spent Wash Tank	500 gal PP	Chemtainer, TC5264CB / Natiional Tank DOPT0500			$1,060			09/07/07	SS	received
SW	2	SW Tank Stand		Chemtainer, CK-CM5264CB Stand							JC	on hand
SW	3	Mixer for SW Tank		Lightning Mod. EV5P25 120v							JC	on hand
SW	4	Blade for SW Tank Mixer		Lightnin, A-310							JC	on hand
SW	5	Spent Wash Pump		Teel, 1P701B centrifugal, 3/4 hp, 115 v 1P, 18 gpm @ 50' hd & 30 gpm @ 40' hd							JC	on hand
SW	6	SW Electrical Heater		SRNL Fabricated ST-MDX5-9878							DF	on hand
		* Organization or initials of responsible person in SRNL. Note that the initials will be updated as activity changes, e.g. design, specifications, procurement, inspection etc.										
		* **Initials**: DF-Don Fisher, JC-Jerry Corbett, JG-John Gordon, MR-Mike Restivo, MW-Mike Williams, SS-Susan Shouse, TS-Tim Steeper, ZQ-Zafar Qureshi										
		Note: This list matches P&ID Revision B.										

APPENDIX 8.9

READINESS CERTIFICATION DOCUMENT
(Consisting of 11 pages
Including coversheet)

WASHINGTON SAVANNAH RIVER COMPANY
Savannah River National Laboratory
Engineering Development Laboratory

Fractional Crystallization Pilot Plant Readiness Certification

April 2008

Prepared by: _____ 4/16/08
Biff Giddings Date
Manager, Engineering Development Laboratory

Reviewed by: _____ 4-15-08
John C. Miller Date
Manager, SRNL Safety Programs

Approved by: _____ 4/15/08
Jeffrey C. Griffin Date
Manager, Research Programs, E&CPT/SRNL

Introduction

Fractional crystallization has been studied as a potential Hanford tank waste pretreatment process since January 2005. An extensive program of laboratory glassware (bench-scale) testing was performed on simulated and actual wastes to quantify the characteristics of the crystallized products and determine their correlation to thermodynamic model predictions (see reference 1)

The Engineering Development Laboratory (EDL) of SRNL assembled a Test Facility to achieve test conditions specified in the Process Flow Diagram (PFD) developed by AREVA. Major components such as Crystallizer Vessel, Reboiler, Circulation Pump and Centrifuge were provided by Swenson Technology Inc. EDL designed and fabricated balance of the system based on these documents and fundamental knowledge and experiences established in previous related experimentation.

The final design was documented by EDL researchers and is shown in the established P&ID (see reference 2). This document was used to fabricate and install the components that will be utilized during the testing. The fabrication and installation was completed by the Engineering Development Laboratory Technicians and work was supplemented utilizing Bechtel Construction Pipefitters and Laborers as obtained through the existing site Memorandum of Understanding (MOU) process. Construction was started in August 2007 and completed in February 2008. The acceptance of construction was completed as specified in the Construction Acceptance Plan for the Facility (see reference 3).

Following construction activities, the facility initiated a series of system checks and functional tests commensurate with the design of the facility and with regards for the safety inspection requirements for the facility and equipment. All primary functional testing has been completed and the system is ready for chemical operations

Purpose

The purpose of this report is to establish a documentation record for the readiness of the facility for chemical operations based on the following criteria:

Work Construction
Safety Compliance Documentation and Controls
Compliance with Conduct of Research and Development
Work Documentation Completion and Readiness
Training Status for Personnel
Compliance with Expectations from Customer
Satisfying Internal Management Requirements
Satisfying DOE Oversight

Scope

The Fractional Crystallization Pilot Test Facility is Research and Development process that was conceived, built and tested per established procedures. Operations of the facility are planned within the constraints of the established Conduct of Research and Development Manual requirements. The readiness assessment for the operations of the facility was performed commensurate with the hazard category of the facility and a graded approach was taken to ensure the proper level of oversight and review was performed prior to each phase of installation and testing.

This document establishes the compliance with established procedures as defined above and provides a summary report of readiness to proceed with prescribed chemical operations as defined by established test documentation.

Methodology

The process used to establish the readiness for operations included the review of supporting documentation, facility walk-downs, witnessing testing and training activities and interviews with key personnel.

Analysis

The project testing program detailed in RPP-PLAN-34134, *Hanford Medium/Low Curie Waste Pretreatment Alternatives Project Integrated Test and Demonstration Plan* (ITDP) defines the approach for testing of proposed alternatives to address waste treatment needs at the River Protection Project (RPP) at the Hanford Site facilities. The end result of the testing program will provide the information that will be used to confirm the process model, validate design assumptions, resolve uncertainties and provide sufficient information to support initiation of a conceptual design following a CD-0 approval (DOE Order 413.3A). See the ITDP for details.

An extensive program of laboratory glassware (bench-scale) testing was performed on simulated and actual wastes to quantify the characteristics of the crystallized products and determine their correlation to thermodynamic model predictions. Reports on this testing program (RPP-RPT-27239, *Hanford Medium/Low Curie Waste Pretreatment Project – Phase I Laboratory Report,* RPP-RPT-30905, *Fractional Crystallization Simulant Test Comparisons,* and RPP-RPT-31352, *Fractional Crystallization Flowsheet Tests with Actual Tank Waste*) contain descriptions of the waste historical background, crystallization process theory, agreement with model predictions, and effectiveness of the FC process in meeting project performance requirements.

With the conceptual process established, the Fractional Crystallization Pilot Test Facility process was developed. Base component/equipment and conceptual pilot plant design documents were provided to SRNL Task Technical Process and included Process Flow Diagrams, system Block Flow Diagrams, Crystallizer design documentation and operating instructions, Centrifuge design documentation and functional design specifications for control systems and proposed pilot plant test plans for the sequential testing of the facility. All of these documents can be obtained from the EDL Principal Investigator (PI) assigned to the task.

The supplied design and plan documentation were utilized by EDL personnel to establish a foundation for how testing would be accomplished at the EDL facility at SRNL. The analysis of readiness is shown below as previously defined in the purpose section of this report.

I. Work Construction

A baseline P&ID was developed based on documentation provided by the customer. This P&ID was utilized as the guiding document for the installation of equipment to be utilized in the pilot test facility. Personnel (Technician and Researcher) skills, previously established within the facility, were utilized to construct the test facility based on the design document. Construction personnel were added to the workforce to supplement the existing technicians used for the installation of equipment as designed. A facility construction schedule was developed and status was provided twice weekly to ensure facility construction and inspection testing were accomplished within defined parameters and prescribed timeframes. A weekly status report was provided to the Hanford customer and this was used as a means of refining plant design needs and developing action items to resolve technical and programmatic challenges encountered in progress. A detailed schedule can be obtained from the SRNL Program Controls group, as desired, to show the level of effort that went into the construction of the facility. A construction acceptance plan was written to ensure construction was accomplished as defined and tested in accordance with plans (see reference 3).

II. Safety Compliance Documentation and Controls

Various levels of safety documentation were established in conjunction with the construction and testing of the facility.

A preconstruction Process Hazard Review (PHR) (see reference 4) was completed to define the technical "What-If" scenarios for the facility. This information was used to refine design documents and facility equipment installation. Twenty one (21) items were identified in this initial PHR. Many of these items were corrected during the construction of the facility.

A follow-up Pre-operations Process Hazard Review (PHR) was conducted following construction. This resulted in an expansion of the original "What-If" list (from preconstruction) to a total of thirty four (34) items. All items have been addressed/resolved from the two PHR assessments.

Several Job Hazard Analysis (JHA) documents were developed in support of the Fractional Crystallization Process as follows:

- SRNL-PSE-2008-00007, Installation of Fiberglass Insulation on Piping and Instruments
- SRNL-PSE-2008-00035, Use of Frothpak Foam Process for Tank Bottom Support
- SRNL-PSE-2008-00041, Fractional Crystallization Acetone Washing of Crystals in 786-A
- SRNL-PSE-2008-00044, Fractional Crystallization Sampling and Sample Preparations
- SRNL-PSE-2008-00057, Fractional Crystallization Operations

The Safety Compliance process involved several layers of safety inspections per Manual 8Q, procedure 51 Final Inspection Acceptance (FAI-51) requirements. These inspections involved:

- Steam Generator Installation [Seven (7) items identified and correct]
- Fractional Crystallization Operations [Eight (8) items identified and 7 items corrected, one item remaining dealing with specific Spill Kit materials, which have been ordered to supplement existing spill control materials in the facility]
- Fractional Crystallization Circulation and Draw-off Pump Equipment Installation [Three (3) items identified and corrected]
- Installation of Centrifuge Equipment [Four (4) items identified and corrected]

III. Compliance with Conduct of Research and Development

Per the requirements of the Conduct of R&D manual, a Hazards Assessment Package (HAP) was developed and documented as SRNL-PSE-2007-0022 for the Fractional Crystallization process. From this review, specific precautions and documentation were addressed as follows:

- Chemical usage, including a caustic corrosive simulant; acetone used for sampling to lock in crystals, Sodium Chromate as an additive to match Hanford Waste tanks
- Steam hazards and controls
- Environmental Evaluation Checklist #TC-A-2007-007
- Confined Space issues inside Crystallizer
- JHA/PHR requirements
- Hazardous Energy (steam)
- Management of Safety Basis (MSB) review

In addition, two sampling HAPs were developed for the process:

- SRNL-PSE-2007-0265, Fractional Crystallization Sampling at 786-A
- SRNL-PSE-2007-0274, Fractional Crystallization Pre-Run Sampling

As previously described, the PHR process was followed to review the additional safety requirements for operation.

Also, as previously described, Job Hazards Analysis documents were developed per the requirements of the HAPs.

IV. Work Documentation Completion and Readiness

The requirements of SRNL Manual L1, procedure 1.01, Administration of SRNL Procedures and R&D Work Control Documents, were reviewed and it was determined that the operations will be performed through the use of work instructions. Equipment installation and testing was performed using R&D directions and technician/researcher training and education.

The Work Instructions developed in support of Fractional Crystallization Pilot Test Facility are as follows:

- ITS-WI-0028, Fractional Crystallization Test Facility. This is the main operations and abnormal operations procedure. This procedure also references the Test Run Plan for performance of testing of the facility and required expected results.
- ITS-WI-0023, Fractional Crystallization – Apparent Settling Volume
- ITS-WI-0024, Fractional Crystallization – Turbidity Measurements
- ITS-WI-0025, Fractional Crystallization – Crystal Size Distribution by Dry Sieving
- ITS-WI-0026, Fractional Crystallization – Sampling and Sample Preparation
- ITS-WI-0029, Fractional Crystallization – Weight Percent Solids and Density Measurement

The sampling documents have been developed, reviewed and approved for use. The main operating instruction has recently been revised, per the Validation and Verification process and approved.

V. Training Status for Personnel

The training of personnel associated with Fractional Crystallization has been performed using a variety of techniques as discussed below. Three of the four assigned shift technicians were involved in the construction of the facility and the subsequent testing (hydrostatic, pressure, circulation) as defined by work instructions or researchers directions. One technician had been partially involved with construction and has been involved with testing.

All technicians and test engineers have been trained on the following:

- Centrifuge Training LSEDL001
- System Overview LSEDL003
- ConOps for R&D LSEDL002
- Sample Management, based on researchers demonstration on sampling and techniques. This was done as a Read-and-Sign process.
- Two Sessions were held with the Test Engineers and Technicians to demonstrate the sample processing and analysis required during shift operations.

In addition, the technicians had Practical Factor demonstrated skills documented for the following items:

- Operations PracFac LSED0004
- Abnormal Operations PracFac LSED005

Each of the test engineers and technicians have been paired up to become familiar with the operating instructions, walked down the procedure and all teams provided comments to the Principal Investigator for revision. These walk downs were used as the Verification and Validation of the existing procedure for operations.

At various phases of system boilout, the technicians and test engineers were involved with the operations of the key components, including the operations of the steam control valve, which is used as the shutdown mechanism in most of the abnormal operations expected to be encountered in the performance of testing.

VI. Compliance with Expectations from Customer

The Hanford (CH2MHill) and AREVA customers have been involved in all aspects of the installation/construction and program reviews. Input has been sought and received for the following items associated with the Fractional Crystallization System:

- Preconstruction Process Hazard Review
- Preoperational Process Hazards Review
- Preparedness Assessment Review
- Sample/Analytical Management Plan
- Task Technical and Quality Assurance Plan
- Operations Work Instruction
- Test Run Plan

In addition, the customers provided feedback on the Process and Instrumentation Drawing (P&ID), the Instrument listing, the Valve listing and the Material listing.

Weekly teleconference meetings where held between SRNL and the customers to cover the construction and pre-operational testing schedule and to address various technical issues that required resolution by SRNL or by the customer. Over 90 action items were developed and received a disposition on the action item list.

The SRNL DOE Facility Representatives have been involved in the Fractional Crystallization process at various levels. Information has been provided to the representatives concerning schedule progress, facility operations documentation and training. The Facility Representatives have attended the initial classroom training for Centrifuge Operations, Fractional Crystallization Operations and Conduct of Operations for Research and Development Training. Feedback was obtained from the DOE customer on the operating Procedure, Conduct of Operations and System Status controls.

VII. Shift Operations and Facility Control:

The operation of the Fractional Crystallization system is under the control of the Test Engineers who will act as the shift managers for the facility. Shift activities include the following:

- A detailed shift turnover using the established turnover checklist
- A daily pre-job briefing each shift, to include the Shift Test Engineer and Technician.
- A review of the Status Board for the facility
- Assurance that minimum staffing is met (1–STE and 1-Technician)
- Daily round sheet completion
- Logbook maintenance
- Test Run Plan review and compliance

VIII. Span of Command and Control:

The Test Engineers are responsible for the operations of the facility. An expectation meeting was held with the four Shift Test Engineers and it was clarified that they were the sole control for the testing performed in the facility. All test issues, plant configuration and any other concerns are to be processed and controlled by the Shift Test Engineers. This includes interfaces with EDL researchers and Hanford Customer interfacing. Technician directions are given via the Shift Test Engineer and the approved work instruction/test plan.

IX. Abnormal Operations:

The existing work instruction is written with twelve (12) sections of abnormal operations controls. Any activities or incidents that are not directly covered by these sections will require a level of analysis by the Shift Test Engineer to ensure any response is appropriate. During the expectation meeting with the Shift Test Engineers, it was clarified that we will not troubleshoot significant activities on the spot. In some cases a written plan will need to be developed to ensure we respond appropriately. No changes to the existing procedure and test run plan will be made without review and approval by the PI, Manager of EDL and other members of the established Joint Test Group as defined in the Task Technical and Quality Assurance Plan (TT&QAP).

A protocol was established with the SRNL Research Operations Department (ROD) defining the expected interfaces with the facility during emergency and abnormal conditions. Facility support for SIRIM/ORPS issues remains with ROD. All facility issues will be communicated to ROD for information and response as necessary. The ROD control room will be contacted for assistance to emergency conditions, including personnel injuries and assistance with abnormal conditions related to chemical management.

Equipment Maintenance:

Because of the short duration of the pilot test (4-6 weeks) there is no established preventative maintenance planned during testing. Equipment repairs will be made in accordance with established facility hazard assessment and controls documentation or additional hazards assessment and work controls documentation will be provided. Critical Spare Parts were reviewed by the facility and key components are on hand and this was agreed upon by the customer. High dollar items, such as the main recirculation pumps, do not have replacements on hand and will cause the suspension of operations if problems occur and cannot be readily resolved.

References

1. RPP-PLAN-34135, *Hanford Medium/Low Curie Waste Pretreatment Alternatives Project Fractional Crystallization Pilot Plant Test Plan*, August 2007, Rev. 0

2. SRNL Drawing: EES-23164-M6-001, *Fractional Crystallization Pilot Scale Facility, P&ID*, March 2008, Rev M44

3. WSRC-TR-2007-00485, *Hanford Medium/Low Curie Waste Pretreatment Alternatives Project Fractional Crystallization Pilot Scale Testing, Construction Acceptance Plan*, January 2008, Rev 0

4. X-PHR-A-00003, *Pre Construction Process Hazards Review for the Fractional Crystallization Process Test Facility*, November 2007, Rev 0.

5. X-PHR-A-00003, *Pre Operations Process Hazards Review for the Fractional Crystallization Process Test Facility*, March 2008, Rev 1.

6. ITS-WI-0028, *Fractional Crystallization Test Facility–Work Instructions,* April 2008, Rev. 0

7. WSRC-TDP-2008-A-00001, *Task Technical and Quality Assurance Plan for Fractional Crystallization Pilot Scale Testing*, March 2008, Rev. 0.

8. SRNL-PSE-2008-00041, *Job Hazards Analysis for Fractional Crystallization Acetone Washing of Crystals in 786-A*, February, 2008, Rev. 0

9. SRNL-PSE-2007-00265, Conduct of R&D-ISM Hazards Assessment Package (HAP) for Fractional Crystallization Sampling in 786-A

10. EEC TC-A-2007-071, *Environmental Evaluation Checklist–Fractional Crystallization (FC) Pilot Scale Test*, Rev. 0

11. SRNL-PSE-2007-00242, *JHA Troubleshooting Equipment for Fractional Crystallization*, March 2008, Rev. 0.

12. SRNL-PSE-2008-0046, *Analytical Plan for Fractional Crystallization Pilot Plant*, April 2008, Rev. 0

Fractional Crystallization Readiness Checklist for Operations

Checklist Action Item	Status Complete (Y/N)	Comments
1) Statement of Work	Y	On file with the PI
2) Design Documentation	Y	Areva,Swenson and KMPF designed documents were obtained and utilized in the development of the P&ID
3) Pre-Construction Process Hazards Review (PHR)	Y	All issues were addressed
4) Hazards Assessment Package (HAP)	Y	On file with the PI
5) Construction Schedule Developed	Y	On file with the PI
6) Job Hazards Assessment (JHA)	Y	Completed and on file with FLM in 786-A
7) Environmental Evaluation Checklist (EEC)	Y	On file with PI
8) Final Acceptance Inspection (FAI-51) per Manual 8Q, procedure 51	Y	Completed and all issues addressed, awaiting one spill kit
9) Pre-operational Process Hazards Review (PHR)	Y	Completed and all issues have been addressed
10) Preparedness Assessment Review	Y	Completed and no operational issues identified
11) Task Technical and Quality Assurance Plan (TT&QAP)	Y	On file with PI
12) Work Instructions/Procedures	Y	Issued following V&V
13) Sample/Analytical Plan	Y	On file with PI
14) Instrumentation Checkout	Y	On file with PI
15) Equipment Checkout	Y	On file with PI
16) Water-Run Testing	Y	Initial runs completed, additional run plan for 4/10/08
17) Chemicals Ordered, Received and Inventoried	Y	All base simulant received, modification simulant on order
18) Operations Training	Y	All training completed with exception to the Pracfacs
19) Abnormal Operations Training	Y	All training completed with exception to the Pracfacs
20) Sample Management Training	Y	Completed as of 4/9/08

APPENDIX 8.10

FINAL BENCHMARK TEST RUN PLAN
(Consisting of 3 pages
Including coversheet)

Test Run Plan

Test Run Plan No. FC-TRP-2008-01A

Date: 4/27/2008

1. Title. Test 1A. Benchmark Testing (Repeat)

2. Test Objectives: A. Startup and fill the pilot system
B. Achieve steady state operations
C. Produce acceptable crystals
D. Establish normal operating parameters
E. Check the correlation between the PFD and the pilot operations

3. Test Conditions/Parameters: *Note the values given below are under steady state operations. During the initial start up, the will vary.*

1. Feed Chemistry: SST Early Feed Phase 2 simulant (without cesium and chromium)
2. Crystallizer Vessel Liquid Operating Level 50% (50% default value=15" above draft tube top, 0%=9", 100%=21")
3. Reboiler Steam Flow Rate, lbm/min. 11.0
4. Crystallizer feed flow rate, gpm (under manual mode): 1.5 gpm
5. Number of Draft Tube Extensions: None
6. Crystallizer Pressure in the vapor space (psia): 0.8 – 1 psia **(adjust this pressure to achieve 131F Crystallizer temperature)**
7. Crystallizer Temperature: 131F
8. Circulation Pump Flow Rate (gpm): 600 gpm
9. Process Tanks Desired Temperature, F 131F
10. PD Tank contents:
    Initial: 250 gal DI H2O Steady State: 300 gal, salt solution Sp. Gr. 1.46
11. RW Tank contents:
    Initial: 0 gal Steady State: 300 gal, recycled wash Sp Gr 1.45
12. SW Tank contents:
    Initial: 0 gal Steady State: 300 gal, recycled wash Sp Gr 1.43
13. Condensate Tank
    Initial: 250 gal DI H2O Steady State: 250 gal condensate

14. Initial Feed Tank B (Note: for the Benchmark Tests, the initial feed sequence is B – A – C - D, then the feed sequence is always B – C – D – B – C –D with reconstituted feed)
15. Centrifuge Settings:

    As recommended by the Customer CH2MHILL. **(Note: The Centrifuge settings will be varied based on cake properties to achieve optimum performance)**

4. Analytical Samples

   Draw samples per sample plan. The actual samples schedule will be controlled by the sampling computer next to the fume hood.

5. System alignment modifications from Work Instructions:

1.	Condensate flow rate to Spent Wash Purge, gpm (FT CO18)	0.29 gpm
2.	Condensate flow rate to PD product, gpm (FT CO24)	0.56 gpm
3.	Spent wash recycle flow rate to Crystallizer, gpm (FT SW09)	1.32 gpm
4.	Spent wash Purge flow rate to Tank E, gpm (FT SW06)	0.29 gpm
5.	Product dissolver flow to FR Tanks (B, C or D), gpm (FT PD09)	0.53 gpm

6. Special equipment or instruments: N/A

7. Precautions
   - Centrifuge – Do not exceed 3000 rpm during operation
   - Reboiler – Do not exceed a slurry temperature rise of greater than 3F across the reboiler during heat up or operation
   - Crystallizer – Do not allow the liquid level in the crystallizer to go outside the range of 20% - 80%.

8. References:
   - FC Pilot Test Plan – RPP-PLAN-34135, Rev 0
   - SRNL P&ID – EES-23164-M6-001, Rev 61
   - SRNL FC Work Instructions – ITS-WI-0028
   - SRNL Pilot Plant Analytical Plan – SRNL-PSE-2008-00046
   - Equipment Vendor Manuals
   - AREVA Drawing C-0110-008 Rev 1, Process Flow Diagram

9. Acceptance Criteria
   - The filter cake dewaters, dislodges and dissolves easily.
   - A feed rate of 1.5 gpm is achieved without overloading the centrifuge.
   - The system's controls are stable in full automatic operation.

APPENDIX 8.11

FINAL BASELINE TEST RUN PLAN
(Consisting of 3 pages
Including coversheet)

Test Run Plan

Test Run Plan No. FC-TRP-2008-02

Date: 5/20/2008

1. Title. Test 2 Baseline Testing

2. Test Objectives:
A. To determine DF for cesium
B. Achieve steady state operations
C. Produce acceptable crystals
D. Establish normal operating parameters
E. Check the correlation between the PFD and the pilot operations

3. Test Conditions/Parameters: *Note the values given below are under steady state operations. During the initial start up, the will vary.*

16. Feed Chemistry: SST Early Feed Phase 2 simulant with cesium
17. Crystallizer Vessel Liquid Operating Level 50% (50% default value=15" above draft tube top, 0%=9", 100%=21")
18. Reboiler Steam Flow Rate, lbm/min. 11.0
19. Crystallizer feed flow rate, gpm (under manual mode): 1.5 gpm
20. Number of Draft Tube Extensions: None
21. Crystallizer Pressure in the vapor space (psia): 0.8 – 1 psia **(adjust this pressure to achieve 131F Crystallizer temperature)**
22. Crystallizer Temperature: 131F
23. Circulation Pump Flow Rate (gpm): 600 gpm
24. Process Tanks Desired Temperature, F 131F
25. PD Tank contents:
    Initial: From Benchmark Test Steady State: 300 gal, salt solution Sp. Gr. 1.46
26. RW Tank contents:
    Initial: From Benchmark Test Steady State: 300 gal, recycled wash Sp Gr 1.45
27. SW Tank contents:
    Initial: From Benchmark Test Steady State: 300 gal, recycled wash Sp Gr 1.43
28. Condensate Tank
    Initial: 250 gal DI H2O Steady State: 250 gal condensate

29. Initial Feed Tank B (Note: for the Baseline Tests, the initial feed sequence is B – A – C - D, then the feed sequence is always B – C – D – B – C –D with reconstituted feed)
30. Centrifuge Settings:

    As recommended by the Customer CH2MHILL. **(Note: The Centrifuge settings will be varied based on cake properties to achieve optimum performance)**

4. Analytical Samples

   Draw samples per sample plan. The actual samples schedule will be controlled by the sampling computer next to the fume hood.

5. System alignment modifications from Work Instructions:

6.	Condensate flow rate to Spent Wash Purge, gpm (FT CO18)	0.29 gpm
7.	Condensate flow rate to PD product, gpm (FT CO24)	0.56 gpm
8.	Spent wash recycle flow rate to Crystallizer, gpm (FT SW09)	1.32 gpm
9.	Spent wash Purge flow rate to Tank E, gpm (FT SW06)	0.29 gpm
10.	Product dissolver flow to FR Tanks (B, C or D), gpm (FT PD09)	0.53 gpm

6. Special equipment or instruments: N/A

7. Precautions
   - Centrifuge – Do not exceed 3000 rpm during operation
   - Reboiler – Do not exceed a slurry temperature rise of greater than 3F across the reboiler during heat up or operation
   - Crystallizer – Do not allow the liquid level in the crystallizer to go outside the range of 20% - 80%.

8. References:
   - FC Pilot Test Plan – RPP-PLAN-34135, Rev 0
   - SRNL P&ID – EES-23164-M6-001, Rev 61
   - SRNL FC Work Instructions – ITS-WI-0028
   - SRNL Pilot Plant Analytical Plan – SRNL-PSE-2008-00046
   - Equipment Vendor Manuals
   - AREVA Drawing C-0110-008 Rev 1, Process Flow Diagram

9. Acceptance Criteria
   - The filter cake dewaters, dislodges and dissolves easily.
   - A feed rate of 1.5 gpm is achieved without overloading the centrifuge.
   - The system's controls are stable in full automatic operation.

APPENDIX 8.12

PILOT TESTING TIME LINE
(Consisting of 3 pages
Including coversheet)

Date	Day	Time	Event	Feed Tank
2/11/2008			Crystallizer vacuum test with mechanical pump was completed successfully	
3/19/2008			Crystallizer vacuum test with steam jet pump was completed successfully	
3/27/2008			**Water boilout test was started**	
4/4/2008			Centrifuge knife was photographed and characterized dimensionally	
4/15/2008	Tue	12:00	**Water boilout test was completed**	
		16:41	**Benchmark Tests started.**	
		20:01	Initial fill of Crystallizer from Tank B completed (910 gallons transferred)	B
4/16/2008	Wed	9:54	Started steam supply to reboiler	
		20:38	Steam Generator #2 shut down for burning odor (one of the 6 heating elements failed)	
4/17/2008	Thu	6:43	Feed Pump FR PUMP1 discharge fitting leaked ~260 gallons in to secondary containment	
		14:00	System restarted after pump repairs	
4/18/2008	Fri	16:57	Switched feed from Tank B to Tank A	A
		22:44	Slurry density 1.67 g/ml (from pulled sample)	
4/19/2008	Sat	7:30	**Centrifuge started**	
		10:12	First batch of crystals harvested, centrifuge operated at 1360 rpm.	
		11:00	Centrifuge basket full of crystals. Centrifuge bogged down. Cleared chute and performed 2 heel removals	
		11:54	Centrifuge speed increased to 1825 rpm and throttled down the feed valve (too much feed to the centrifuge)	
		14:30	Centrifuge speed increased to 2500 rpm, ran 4 cycles and then increased to 2700 rpm	
		15:32	Centrifuge speed increased to 2800 rpm, ran 4 cycles and 2 heel removals	
		16:37	Centrifuge speed decreased to 2700 rpm, ran 7 cycles	
		17:31	Centrifuge speed decreased to 2600 rpm, added heel removal step after every 5 cycles	
		18:30	Installed a double plate type pinch to the feed line to the centrifuge. A similar pinch was applied to the centrifuge discharge port 5 to divert discharge to clear out p-trap connected to port 8	
		19:05	Flexible hose to port 5 bursted due to high pressure as the pinch was increased. Centrifuge emergency stop.	
		19:10	System shutdown started. Diluted crystallizer contents and transferred 997 gal to B and 990 gal to A.	Feed stopped
4/20/2008	Sun		System shutdown due to hose burst	Feed stopped
4/21/2008	Mon		System shutdown due to hose burst	Feed stopped
4/22/2008	Tue		System modifications comleted. New SL18B valve added.	Feed stopped
4/23/2008	Wed		System vacuum tested	Feed stopped
4/24/2008	Thu		Contents of Tank A mixed	Feed stopped

Date	Day	Time	Event	Feed Tank
4/25/2008	Fri	0:35	Transferred 835 gallons of feed from Tank A to Tank E.	Feed stopped
		3:30	Transferred 880 gallons of feed from Tank B to Tank A	Feed stopped
		4:27	Transferred 850 gallons of feed from Tank E to Tank B	Feed stopped
		5:00	Mixing of Tanks A & B complete	Feed stopped
		8:00	Preparations to fill the crystallizer started (to be filled under vacuum)	Feed stopped
		9:43	Crystallizer filled to the operating level by transferring 970 gallons from Tank B.	Feed stopped
		13:34	Started steam supply to reboiler	Feed stopped
		15:15	Crystallizer operating under normal conditions.	Feed stopped
		15:23	Started feeding crystallizer from Tank B	B
		19:01	Centrifuge heel removal and cleanup complete.	
		21:04	Started filling PD Tank with condensate	
4/26/2008	Sat	6:30	PD Tank temperature not increasing, started work on PD Tank heater control	
		8:29	Switched feed from Tank B to Tank A	A
4/27/2008	Sun		Work on PD Tank heater control continued	
4/28/2008	Mon	15:00	Started centrifuge operation at 2600 rpm	
		18:15	Centrifuge shut down to install a flow restriction (3/8" dia hole) in the feed hose.	
4/29/2008	Tue	9:05	Centrifuge started at 2600 rpm.	
		12:04	Centrifuge speed increased to 2800 rpm	
			Centrifuge tripped several times due to high vibrations	
		19:40	Centrifuge rpm increase to 2700, vibrations acceptable (<4)	
		20:20	Started Spent Wash Tank.	
		22:15	Started centrifuge operation under auto mode	
4/30/2008	Wed	8:15	Recycle Wash Tank heater fuses replaced.	
		8:30	DAS was inadvertently unplugged. DAS was restarted.	
		16:17	Started spent wash flow to Tank E at 0.5 gpm.	
		19:47	Started Spent Wash recycle to the crystallizer at 0.4 gpm. No flow, line was plugged.	
			The centrifuge was operated off and on due to pluggage after peel steps. Started heel removal after 5 cycles.	
5/1/2008	Thu		Continued centrifuge operations off and on due to high vibrations during peel steps	
		17:19	Crystallizer density was found to be too high (1.75 g/ml). Added condensate to bring it below 1.7	
			Continued centrifuge operations off and on due to high vibrations during peel steps	
		19:21	Transferred 620 gallons from Tank E to Tank A.	
		21:50	Started Wash 2 step for centrifuge. No wash 2 flow due to pluggage	
		23:35	Wash 2 flow observed (~0.6 gpm)	
5/2/2008				